STANDARD OPERATING PROCEDURE

INORGANIC CHEMISTRY DATA VALIDATION
2019

TREC, Inc.
A WOODARD & CURRAN COMPANY

March 18, 2019

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SIGN-OFF PAGE

TREC, Inc. Standard Operating Procedure for Validation of Inorganic Analytical Data
Revision 0

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DISTRIBUTION LIST

Standard Operating Procedure – Inorganic Chemistry Data Validation, 2019

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REVISION SUMMARY

Revision No.	Author	Version	Description	Date
0	TREC, Inc., JJ	1	SOP Validation of Inorganic Chemistry Data for CFRSSI 2014, aligns with Jan 2010 NFG	1st QTR 2014
1	TREC, Inc., JJ	2	SOP Validation of Inorganic Chemistry Data for CFRSSI 2015, aligns with Jan 2010 NFG	1st QTR 2015
2	TREC, Inc., JJ	3	SOP Validation of Inorganic Chemistry Data for CFRSSI 2016, aligns with Aug 2014 NFG	1st QTR 2016
3	TREC, Inc., JJ	4	SOP Validation of Inorganic Chemistry Data for CFRSSI 2017, aligns with Jan 2017 NFG	1st QTR 2017
4	TREC, Inc., JG	5	SOP Validation of Inorganic Chemistry Data for CFRSSI 2018, aligns with Jan 2017 NFG	1st QTR 2018
5	TREC, Inc. TD	6	SOP for Inorganic Chemistry Data Validation Aligns with method requirements, generally follows 2017 NFG guidance	March 2019

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TREC Data Validation SOP Revision Summary

The 2019 TREC Data Validation Standard Operating Procedure (DV SOP) has undergone revision to provide greater context to the data validation process. While the DV SOP format has undergone extensive changes, the DV SOP lists validation steps in an order very similar to previous TREC DV SOPs.

Analytical Quality Control Sample Limits

Laboratory quality control (QC) sample requirements for analytical methods other than those referenced in the US EPA National Functional Guidelines for Inorganic Superfund Data Review (NFGs) are now included in the DV SOP. This includes EPA 200.7, EPA 200.8, EPA 245.1, and multiple other methods. The limits used are those specified within the analytical methods when available. Otherwise, analytical SOPs from Pace Analytical were relied on. The justification of using Pace SOPs is that Pace must adhere to British Petroleum's (BP) Laboratory Management Program Statement of Work. The Pace limits align with method requirements, and in cases where method requirements are not stated, the Pace limits are more stringent than those which were specified in previous TREC DV SOPs. Laboratory duplicate precision is an exception for some analyses, but it is assumed, and will be verified, that the Pace duplicate precision requirements align with those required by The NELAC Institute.

In addition to revising laboratory quality control sample limits, laboratory QC sample frequency requirements have been specified.

Laboratory and Field Blank Assessment

The method for assessing field and laboratory blank results above the method detection limit has changed. Previously, blanks were assessed in accordance with Table 1. Table 2 displays the updated method of assessing field and laboratory blank results. Note that the revised method treats any blank detection $\leq$ 1.5X the MDL as a non-detect.

Table 1 – TREC Data Validation SOP Previous Blank Assessment Rules

Field Blank Result	Sample Results	Action for Samples
< Adjusted MDL value due to MB contamination	Any	No action
≥ MDL, but ≤ 2x MDL	Non-detect	No action
	≥ MDL, but ≤ CRQL	Qualify results as non-detect at the CRQL value **(U)**
	> CRQL	No action

Field Blank Result	Sample Results	Action for Samples
> 2x MDL	Non-detect	No action
	≥ MDL, but ≤ 10x blank value	Qualify results as estimated high **(J+)**
	>10x blank value	No action
< 2x (-MDL)	Non-detect	Qualify results as estimated **(UJ)**
	≤ 5x absolute blank value	Qualify results as estimated low **(J-)**

Table 2 - TREC 2019 Data Validation SOP Revised Blank Assessment Rules

Lab Blank Result	Sample Results	Action for Samples
≥ MDL, but ≤ 1.5x MDL	Non-detect	No action
	> MDL, but ≤ RL	No action
	> RL	No action
> 1.5x MDL	Non-detect	No action
	≥ MDL, but ≤ RL	Qualify results as estimated non-detect (UJ)
	> RL, but ≤ 10x blank	Qualify results as estimated high (J+)
	>10x blank value	No action
> RL	See action above for 1.5x MDL value. If any sample results are > MDL and < 10x the blank value, note in the Data Validation Summary that the laboratory failed to re-digest (if applicable) and reanalyze the affected samples. Reanalysis is not required for sample results < MDL or > 10X the blank detection.	
< 2x (-MDL)	Non-detect	Qualify results as estimated (UJ)
	≤ 5x absolute blank value	Qualify results as estimated low (J-)
Lab Sample	**Report Level**	**Frequency[1]**
ICB	L3, L4	At beginning of analytical run, immediately after ICV
CCB	L3, L4	One in every 10 samples, immediately after CCV
MB	L2, L3, L4	One per batch of 20 or fewer samples

[1]ICB/CCB samples not applicable to gravimetric (solids) analyses

Assessment of Field Blanks Based on Laboratory and Field Duplicate Results

The 2019 data validation SOP has clarified that field collected blanks do not receive qualifications based on either laboratory or field duplicate results. There may be cases where the field blank was used as the parent sample for a laboratory duplicate, and in that case, the field blank would be qualified based on laboratory duplicate precision.

LIST OF ACRONYMS AND ABBREVIATIONS

% D	percent difference
% R	percent recovery
°C	degrees Celsius
1.5X	1.5 times
10X	10 times
2X	2 times
5X	5 times
ACM	Anaconda Mining Company
AES	atomic emission spectrometry
AMU	atomic mass unit
As	arsenic
ASTM	American Society of Testing and Materials
Ba	barium
BP	British Petroleum
BPSOU	Butte Priority Soils Operable Unit
CCB	cross contamination blank
CCB	continuing calibration blank
CCV	continuing calibration verification
Cd	cadmium
Cl	chloride
COC	chain of custody
COD	chemical oxygen demand
Cr	chromium
CRDL	contract required detection limit
Cs	cesium
Cu	copper
CVAA	cold vapor atomic absorption
D	duplicate sample
DAR	data assessment report
DF	dilution factor
DI	deionized water
DM	data management
DOC	dissolved organic carbon
DSR	data summary report
DST	data summary table
DV	data validation
DVS	data validation spreadsheet
ECB	equipment contamination blank
F	fluoride
FB	field blank
GW	groundwater

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LIST OF ACRONYMS AND ABBREVIATIONS

GWQC	groundwater quality control sample
HNO3	nitric acid
HT	holding time
I	initial sample result
ICB	initial calibration blank
ICP	inductively coupled plasma
ICV	initial calibration verification
ID	identification
ISC	interference check sample
J	estimated
J-	estimated low
J+	estimated high
L2	level 2 data package
L3	level 3 data package
L4	level 4 data package
LaMP	laboratory management program
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LDS	laboratory duplicate sample
LFB	laboratory fortified blank
LIMS	laboratory information management system
MB	method blank
MDL	method detection limit
Mn	manganese
Mo	molybdenum
MS	matrix spike
MSD	matrix spike duplicate
NH3	ammonia
Ni	nickel
NO2	nitrite
NO3	nitrate
ORP	oxidation reduction potential
Pb	lead
PDS	post digestion spike
QAPP	quality assurance project plan
QC	quality control
R	rejected
Rb	rubidium
RB	rinsate blank
RL	reporting level
RSD	relative standard deviation

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LIST OF ACRONYMS AND ABBREVIATIONS

S	serial dilution result
S	primary sample
SA	spike added
Sb	antimony
SC	specific conductivity
SD	serial dilution
SDG	sample delivery group
SM	standard method
SO4	sulfate
SOP	standard operating procedure
SR	sample result
SSR	spiked sample result
SW	surface water
SWQC	surface water quality control sample
TB	trip blank
TDS	total dissolved solids
TKN	total Kjeldahl nitrogen
TOC	total organic carbon
TSS	total suspended solids
U	non-detect
U	uranium
UJ	estimated non-detect
USEPA	United States Environmental Protection Agency
WO	work order
Zn	zinc
SA	spike added
Sb	antimony
SC	specific conductivity
SD	serial dilution
SDG	sample delivery group
SM	standard method
SO4	sulfate
SOP	standard operating procedure
SR	sample result
SSR	spiked sample result
SW	surface water
SWQC	surface water quality control sample
TB	trip blank
TDS	total dissolved solids
TKN	total Kjeldahl nitrogen
TOC	total organic carbon

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LIST OF ACRONYMS AND ABBREVIATIONS

TSS	total suspended solids
U	non-detect
U	uranium
UJ	estimated non-detect
USEPA	United States Environmental Protection Agency
WO	work order
Zn	zinc

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1.0 PREPARATION

1.1 Review Guidance Documents

The main document that will guide project specific data validation is the applicable project QAPP. Each QAPP contains a table(s) of required laboratory calibration and quality control limits.

BPSOU QAPPS:

BPSOU SW QAPP

File:

Draft_BPSOU_SW_QAPP.pdf

Location:

\\woodardcurran.net\shared\Projects\TREC\9208_AR_MT_BPSOU\9208 - 2009 BPSOU\9208-003_SW-GW_Monitoring\01_SurfaceWater\01_Monitoring_Plans\SW IMP\2019\Submittal\3_4_19\

BPSOU GW QAPP

File:

2019_Mar13DrftQAPP_BPSOU_GW.pdf

Location:

\\woodardcurran.net\shared\Projects\TREC\9208_AR_MT_BPSOU\9208 - 2009 BPSOU\9208-003_SW-GW_Monitoring\02_Groundwater\01_Monitoring_Plans\Monitoring Plan 2019\QAPP\Submittal\2019_03_13\

Rocker data validation follows guidelines in the BPSOU GW QAPP.

Anaconda Copper Mining (ACM) Former Refinery Site:

ACM Site-Wide QAPP

Additional ACM QAPPs can be found at the link below:

ACM Site Document Repository

In addition to the QAPPs, general validation guidance documents can be consulted. Either print a copy of each guidance document listed below or open the documents and refer to an electronic version. Find digital copies of each of these documents here:

Location:

\\woodardcurran.net\shared\Offices\Bozeman\BUTTE\TREC\ARCO\DataValidation\Docs\

CFRSSI Data Validation/Data Management (DV/DM) Plan

File:

CFRSS DVDM Rev2.pdf

CFRSSI DV/DM Plan Addendum

File:

DVDM Plan Addendum.PDF

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The USEPA National Functional Guidelines (NFGs) can be used as a general guidance document; but, be aware that the limits within the NFGs are not necessarily applicable to the analytical methods used for the data that is being validated. This is only a general guidance document.

USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review (January 2017):

File:

USEPA NFG inorganic Jan2017.pdf

BP Laboratory Management Program (LaMP) Technical Requirements (serves as the Statement of Work for BP contract laboratories). This data validation SOP has been written to be in compliance with BP's LaMP Technical Requirements. The BP LaMP Technical Requirements are applicable only to Atlantic Richfield, or BP, data.

File:

BP_LaMP Tech Requirements2017.pdf

1.2 Open Raw Data Deliverable

Open the laboratory report, which is a PDF file that the analytical laboratory provides to the client, or the client's contractor. A Level 2 laboratory report includes a cover letter, sample summary, lab case narrative, sample report forms, lab quality control (QC) results, chain-of-custody, sample receipt forms, and any additional custody documentation (i.e. emails between samplers and the lab). Level 3 reports contain laboratory calibration data and laboratory quality control sample results, as well as preparation logs and analysis run logs. Level 4 reports contain all elements of Level 3 reports and the full analytical run. The name of the laboratory report file matches that of the Sample Delivery Group (SDG) or work order (WO) number. SDG is a unit used to identify groups of samples inclusive under one (or more) Chain-of-Custody (COC). One Data Validation Checklist will be completed for each SDG. Checklists may be Level 2, Level 3, or Level 4, and the checklist templates can be located at the links below.

Level 2 Checklists

File:

SWWWChecklist_template.xlsx

Location:

\\woodardcurran.net\shared\Offices\Bozeman\BUTTE\TREC\ARCO\DataValidation\2019\SWWW\

Level 3 checklist

In development

Level 4 checklist

File:

Level-IV_DV-Checklist_GW.xlsx

Location:

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\\woodardcurran.net\shared\Offices\Bozeman\BUTTE\TREC\ARCO\DataValidation\2019\GW\

Level 2 packages are received for BPSOU base flow monitoring. An example Level 2 data packages is located here:

File:

10421948_frc.pdf

Location:

\\woodardcurran.net\shared\Offices\Bozeman\BUTTE\TREC\ARCO\DataValidation\2018\BF\Raw Data\10421948_Feb\

Several Great Falls projects require Level 3 data packages. An example Level 3 package, for soils analysis, is located here:

File:

10457167_package_L3.pdf

Location:

\\woodardcurran.net\shared\Projects\TREC\3030_AR_MT_GF_Refinery_O&M\OU2\030-Data\05_Soil\03_Raw\Sediment\2018\

Level 4 data packages are required for a subset of BPSOU groundwater sites, all Rocker monitoring, and several Great Falls projects. An example BPSOU Level 4 package is located here:

File:

10429390_package_L4.pdf

Location:

\\woodardcurran.net\shared\Offices\Bozeman\BUTTE\TREC\ARCO\DataValidation\2018\GW\Raw Data\Spring\L4Packages\

Consult with the project managers, or the data management team, to determine the server location of laboratory packages.

Open the Data Validation (DV) Distribution File

Open the data validation distribution. The database dump files are provided to personnel by the database team: Donna Hawley and Jonathan Longden.

The data validation checklist guides the validation process and these checklists are completed as the validator goes through the data packages. Open the appropriate data validation checklist template and save it to the appropriate folder using an intuitive file naming convention.

2.0 HOLDING TIME AND SAMPLE PREPARATION

2.1 Check Holding Times

Check the holding time for each data point. This is performed in Excel by subtracting the sample collection date/time from the sample analysis date/time. You can use the values given in the database dump. Method specific holding times are listed below in Table 1– Holding Times and Preservation

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Requirements for typical analyses. A more thorough list of holding times can be found in the Tables section of this SOP. Note: Because HTs are analyte and method specific, the method of sample analysis must match that listed in the raw data deliverable. If the methods differ, and it is not listed in Table 1, look it up for that specified method Analytical method descriptions are found here:

Location:

\\woodardcurran.net\shared\Offices\Bozeman\TREC Files\Reference_Regs_Specs\EnvironmtlMonitoring\Methods_InorgChem\

Holding times can be found within the methods, and within Pace analytical SOPS, which are located here:

Location:

\\woodardcurran.net\shared\Offices\Bozeman\BUTTE\TREC\ARCO\PaceLabs\Pace SOPs_2018_09\

The file at the link below is a comprehensive list of holding times.

File:

Preservation_Holding_Time.pdf

Location:

\\woodardcurran.net\shared\Offices\Bozeman\BUTTE\TREC\ARCO\DataValidation\Docs\Methods\

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Table 1– Holding Times and Preservation Requirements

Analyte	Method	Holding Time	Preservative	BPSOU BF	BPSOU WW	Diagnostic	Expanded	BPSOU GW	Rocker	Great Falls
Alkalinity: Total, Carbonate, Bicarbonate, & Hydroxide	SM 2320B	14 days	Raw 0-6°C	Total only	Total only			x	x	
Anions by Chromatography (bromide, chloride, fluoride, sulfate)	EPA 300.0	28 days	Raw 0-6°C				Cl, F, SO4	Cl, SO4		x
Anions by Chromatography (orthophosphate-P, nitrate, nitrite)	EPA 300.0	48 hours	Raw 0-6°C							
Chloride	SM4500-Cl C	28 days	Raw 0-6°C						X	
Sulfate	ASTMD 516	28 days	Raw 0-6°C	x	x	x			x	
Dissolved Organic Carbon/Total Organic Carbon (DOC/TOC)	SM 5310 C	28 days	H_2SO_4 < pH 2 0-6°C	DOC	DOC		DOC			
Hardness[1]	SM 2340B	180 days	HNO_3 < pH 2	x	x					
Mercury (aqueous) total and dissolved by CVAA	EPA 245.1,	28 days	HNO_3 < pH 2	245.1	245.1			245.1		

Analyte	Method	Holding Time	Preservative	BPSOU BF	BPSOU WW	Diagnostic	Expanded	BPSOU GW	Rocker	Great Falls
	SW846 7470									
Metals (aqueous) total and dissolved by ICP-AES	EPA 200.7, SW846 6010	180 days	HNO_3 < pH 2				SW846 6010B			
Metals (aqueous) total and dissolved by ICP-MS	EPA 200.8, SW846 6020, 6020A, 6020B,	180 days	HNO_3 < pH 2	200.8	200.8	200.8	SW846 6020A	200.8	200.8	
Metals (aqueous) - Dissolved Exotic by ICP-MS (Cs & Rb)	SW6020 A_E	180 days	HNO_3 < pH 2				x			
Nitrogen - Ammonia	**EPA 350.1** SM 4500-NH3 B/C	28 days	H_2SO_4 < pH 2 0-6°C	x	x					
Nitrogen - NO_2/NO_3	**SM 4500-NO3 H** SM 4500-NO3 E	28 days	H_2SO_4 < pH 2 0-6°C	x	x					

Analyte	Method	Holding Time	Preservative	BPSOU BF	BPSOU WW	Diagnostic	Expanded	BPSOU GW	Rocker	Great Falls
	SM 4500-NO2 B									
Nitrogen - Total Kjeldahl Nitrogen	**EPA 351.2** SM 4500-Norg B	28 days	H_2SO_4 < pH 2 0-6°C	x	x					
pH	EPA 150.1	24 hours	Raw 0-6°C				x	x		
Solids - Total Dissolved Solids	SM 2540C	7 days	Raw 0-6°C	x	x			x		
Solids - Total Suspended Solids	SM 2540D	7 days	Raw 0-6°C	x	x	x				
Specific Conductivity	SM 2510B	28 days	Raw 0-6°C				x	x		
Total Metals in Solids by ICP-MS (Sb, As, Ba, Cd, Cr, Cu, Pb, Mn, Mo, Ni, U, & Zn)	SW6020	180 days	None				x			
Phosphorus - Total/Dissolved	**SM 4500P-B**/E	28 days	H_2SO_4 < pH 2 0-6°C	x	x					

2.2 Assign Data Qualifiers for Exceeded Holding Times

Assign data qualifiers for exceeding holding times using Table 2. With each data qualifier assigned, include reason code "HT" (see narrative below as well as Figure 2). As long as a sample is analyzed by the end of the last day of its recommended holding time, it is considered to be within holding time limits. For example, TSS (7-day holding time) analyzed at 7.9 days is still within the holding time limit.

Use "professional judgment" when assigning data qualifiers based on holding time exceedances. The interpretation of "professional judgment" for the purpose of this SOP is that before a data point is rejected (R), there must be substantial evidence supporting the rejection. For holding times ≤ 14 days, a 2x the recommended holding time limit may be applied. For example, pH (24-hour holding time) analyzed at 47 hours warrants a J- qualifier where pH analyzed at 49 hours warrants an R qualifier. It is rare when 28-day or 180-day holding times are exceeded on our projects, but it does occur. In such instances, the 2x recommended holding time limit is not applicable. You would instead research as to why the holding times were exceeded (sometimes in the case narrative or sample receipt forms) and how the analyte concentration might be affected outside of holding time. If there is no explanation for a holding time exceedance, analyses 10 days to two weeks past the recommended holding time may warrant rejection. For example, total mercury analyzed at 35 days warrants a J- qualifier where total mercury analyzed at 45-days warrants an R qualifier.

Table 2 - Holding Time Action

Holding Time (HT) Result	Action for Samples
< Recommended HT	No Action
> Recommended HT	Qualify results that are ≥ MDL as estimated low (J-) Qualify non-detects as estimated (UJ)
Use professional judgment to determine excessive exceedance of recommended HT.	Qualify all results unusable (R)

Figure 1 Holding Time Action Example

Field Sample ID	Param	Fraction	Result (mg/L)	DF	Lab Flag	DV Qual	Code	Quality	MDL (mg/L)	CRQL (mg/L)	Units	Collection Date	Analysis Date	HT
SWBF0001-012015	TSS	T	0.0074	1				E	0.0012	0.01	mg/L	01/20/2015	01/23/2015	5.61
SWBF0002-012015	TSS	T	0.0035	1				E	0.0012	0.01	mg/L	01/20/2015	01/23/2015	5.59
SWBF0003-012015	TSS	T	0.0017	1	J	J-	HT	E	0.0012	0.01	mg/L	01/20/2015	01/23/2015	7.59
SWBF0004-012015	TSS	T	0.0012	1				E	0.0012	0.01	mg/L	01/20/2015	01/23/2015	5.58

2.1 Verify Proper Sample Preservation

Verify that samples were properly preserved, received at the proper temperature, and filtered as required. You can find this information in the (pdf) raw data deliverable. Information on sample receipt, including preservation requirements are noted by the laboratory on sample receipt forms located after the COC, near the end of the document. Note that dissolved metals, mercury, and dissolved organic carbon samples must be field filtered with a 0.45 µm filter. If field filtering is not possible, preservative should not be added to the sample until it has been filtered.

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2.2 Assign Data Qualifiers for Incorrect Sample Preservation

Assign data qualifiers for incorrect sample preservation using Table 3 - and applying reason code "IP".

Table 3 - Preservation Action

Result	Action for Samples
Aqueous samples received with pH > 2 and pH not adjusted	Qualify results that are ≥ MDL as estimated low **(J-)** Qualify non-detects as estimated (UJ)
Aqueous or soil/sediment samples not maintained at ≤ 6°C (but not frozen) or received >10°C	Qualify results that are ≥ MDL as estimated low **(J-)** Qualify non-detects as estimated (UJ)

3.0 LABORATORY DATA VALIDATION

3.1 Read Laboratory Case Narrative

Read the laboratory cover letter and case narrative found in the (pdf) raw data deliverable. The case narrative will give insight to problems the laboratory had when running analyses (or lack thereof).

3.2 Check Sample ID Numbers

Check the sample ID numbers listed in the raw data report and data dump files. Ensure that they are exactly the same as what is listed on the chain-of-custody, which is found at the end of the (pdf) raw data report. (Upon database import, this check is made automatically for BPSOU and Rocker data.) If there is a discrepancy, the laboratory needs to be notified and submit a revised report with correct sample IDs. The data validator may, alternatively, modify segments of the report by hand to include the correct sample ID numbers. If the report is amended by hand, include a narrative of what was edited in the data validation summary report.

3.3 Verify Laboratory Quality Control (QC) Parameters.

The laboratory must adhere to method requirements for all calibrations and quality control (QC) samples. Calibration steps, calibration limits, and QC sample frequency and limits vary depending on the method. This section first explains laboratory calibration and QC samples and then explains the actions for out-of-compliance calibration or QC samples. Next calibration and QC sample control limits for individual analyses are provided. Several parameters can be analyzed by more than one analytical method, and it is not uncommon for differing methods to have different limits. Thus, within each analytical parameter listed below, more than one method may be listed. The information provided in laboratory reports differs depending on the report level. The tables below indicate which data are reported in Level 1, Level 2, Level 3, and Level 4 reports.

Be aware that samples within a sample delivery group are not necessarily analyzed in a single batch. A laboratory QC batch can consist of up to 20 samples, thus, if more than 20 samples are submitted, samples will be associated with more than one QC batch. Samples within a single SDG may also be broken into more than one batch when fewer than 20 samples are submitted. This is common with total dissolved solids (TDS) and total suspended solids (TSS) analyses. Be certain that validation qualifiers are applied only to samples in the QC batch that is associated with the qualifier.

3.3.1 Instrument Tune

Instrument tuning is applicable only to ICP-MS analyses (SW846 6020 series and EPA 200.8). Prior to calibration, the ICP-MS tuning solution is analyzed. The tune solution contains a range of isotope masses and it establishes instrument accuracy, resolution, and precision prior to calibration. The tune solution must be analyzed five times, consecutively. Any necessary adjustments are made to bring the peak width within the manufacturer's specifications and to adjust the resolution of the mass calibration to within 0.1 atomic mass unit (amu) over a specified amu range. The percent relative standard deviation (RSD) of the absolute signals for all target analytes in the tuning solution must but be $\leq$ 5%. An example tune report is presented in Figure 2. Tune criteria and corrective actions

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Figure 2 – Example Tune Report

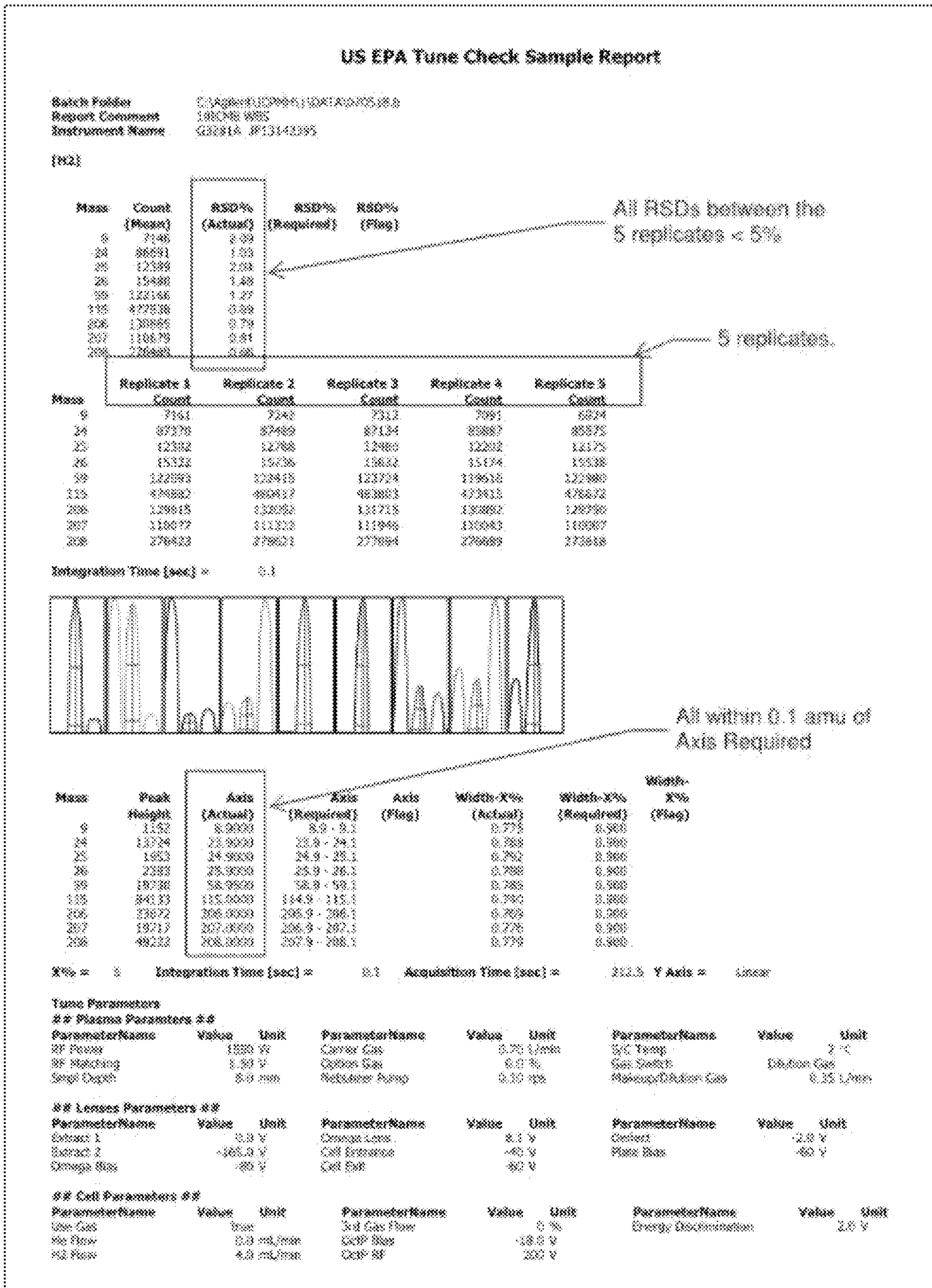

Table 4 – ICP MS Tune Criteria

Calibration Step[1]	Method	Frequency	Control Limits
Tune	SW846 6020 SW846 6020A SW846 6020B EPA 200.8	Prior to calibration	Tune solution analyzed five times, consecutively Mass calibration within 0.1 amu % RSD of absolute signals < 5%

[1]Reported in Level 3 and Level 4 packages

Table 5 – ICP MS Tune Actions

Calibration Step[1]	Method	Tune Result	Action for Samples	Qualifier Code
Instrument Tune	SW846 6020 SW846 6020A SW846 6020B EPA 200.8	Not performed	Qualify all data as unusable (R)	Tune
		5 consecutive analyses of tune solution not performed	Use professional judgement. At a minimum qualify detects as estimated (J) and non-detects as estimated non-detect (UJ)	Tune
		Mass calibration resolution not within 0.1 amu	Qualify detects as estimated (J) Qualify non-detects as estimated non-detect (UJ)	Tune
		> 5% RSD	Qualify detects as estimated (J) Qualify non-detects as estimated non-detect (UJ)	Tune

3.3.2 Laboratory Calibration Data

Calibration data are provided in Level 3 and 4 reports. A calibration curve is established with a blank and various standards. The calibration curve fit is a linear regression of results for the blank and calibration standards. The calibration curve fit can be found at the beginning of the raw data in Level 4 reports only. Table 4 details corrective actions for out of compliance calibration curves.

Initial calibration verification (ICV) and continuing calibration verification (CCV) results are reported as percent recoveries (%R). These are determined by:

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$$\%R = \frac{\boldsymbol{Found\ Value}}{\boldsymbol{True\ Value}}\ \boldsymbol{x\ 100}$$

Figure 3 provides an example of ICV/CCV data reported by the laboratory. Note that several CCVs have > 110% recovery for Ca, Mg, Si, Na, and Zn. If this occurs, consult the analysis run log, which is provided in Figure 4. The out of compliance CCV is highlighted in Figure 4. Five samples were run between the out of compliance CCV and the next in compliance CCV. However, Ca, Mg, Si, Na, and Zn results were not reported from that run sequence, but from a later run sequence. Thus, these sample results should not be qualified.

Figure 3 - ICV/CCV Example from Level 4 Report

FORM II INORGANIC-2
INITIAL AND CONTINUING CALIBRATION VERIFICATION

Lab Name: Pace Analytical - Minnesota SDG No. : 10443691 Contract: Rocker

Initial Calibration Verification Source:

Continuing Calibration Verification Source: 174801

Concentration Units: ug/L Instrument ID: 10ICM3

	Continuing Calibration Verification									
	09/05/2018 00:10			09/05/2018 00:50			09/05/2018 01:29			Control Limit
Analyte	True	Found	%R	True	Found	%R	True	Found	%R	
Arsenic	80	86.8	108.6	80	86.7	108.4	80	86.2	107.8	90-110
Cadmium	80	84.6	105.8	80	84.1	105.1	80	84.0	105.0	90-110
Calcium	1000	1180	117.7	1000	1180	117.5	1000	1130	112.9	90-110
Copper	80	87.0	108.8	80	87.8	109.8	80	86.7	108.4	90-110
Iron	1000	1100	109.5	1000	1070	107.2	1000	1080	107.8	90-110
Lead	80	84.9	106.1	80	84.0	105.0	80	84.0	105.1	90-110
Magnesium	1000	1140	113.7	1000	1110	110.6	1000	1130	112.6	90-110
Manganese	80	87.2	109.0	80	85.8	107.2	80	85.7	107.2	90-110
Potassium	1000	1090	109.3	1000	1080	107.7	1000	1060	106.4	90-110
Silicon	1000	1170	117.4	1000	1100	110.3	1000	1120	112.2	90-110
Sodium	1000	1100	110.5	1000	1100	110.4	1000	1070	107.4	90-110
Zinc	80	88.5	110.6	80	87.9	109.9	80	87.5	109.4	90-110

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Figure 4 – Analysis Run Log Example from Level 4 Report

FORM XIII INORGANIC-1
ANALYSIS RUN LOG

Lab Name: Pace Analytical - Minnesota SDG No. : 10443691 Contract: Rocker

Instrument ID: 10ICM3 Analysis Method: EPA 200.8

Start Date: 09/04/2018 22:03 End Date: 09/05/2018 02:17

Sample Name	Lab Sample ID	D/F	Date	Time	As	Ca	Cd	Cu	Fe	K	Mg	Mn	Na	Pb	Si	Zn
20496315CAL0	20496315CAL0	1	09/04/2018	22:03	X	X	X	X	X	X	X	X	X	X	X	X
20496316CAL1	20496316CAL1	1	09/04/2018	22:06	X	X	X	X	X	X	X	X	X	X	X	X
20496317CAL4	20496317CAL4	1	09/04/2018	22:10	X	X	X	X	X	X	X	X	X	X	X	X
20496318CAL5	20496318CAL5	1	09/04/2018	22:13	X	X	X	X	X	X	X	X	X	X	X	X
20496319CAL6	20496319CAL6	1	09/04/2018	22:17	X	X	X	X	X	X	X	X	X	X	X	X
20496320CAL2	20496320CAL2	1	09/04/2018	22:20	X	X	X	X	X	X	X	X	X	X	X	X
20496321CAL3	20496321CAL3	1	09/04/2018	22:24	X	X	X	X	X	X	X	X	X	X	X	X
20496322ICV	20496322ICV	1	09/04/2018	22:27	X	X	X	X	X	X	X	X	X	X	X	X
20496323ICB	20496323ICB	1	09/04/2018	22:34	X	X	X	X	X	X	X	X	X	X	X	X
20496326ICSA	20496326ICSA	1	09/04/2018	22:44	X	X	X	X	X	X	X	X	X	X	X	X
20496327ICSAB	20496327ICSAB	1	09/04/2018	22:48	X	X	X	X	X	X	X	X	X	X	X	X
20496328CCV	20496328CCV	1	09/04/2018	22:51	X	X	X	X	X	X	X	X	X	X	X	X
20496329CCB	20496329CCB	1	09/04/2018	22:58	X	X	X	X	X	X	X	X	X	X	X	X
20496330CCV	20496330CCV	1	09/04/2018	23:25	X	X	X	X	X	X	X	X	X	X	X	X
20496331CCB	20496331CCB	1	09/04/2018	23:29	X	X	X	X	X	X	X	X	X	X	X	X
20496332CRDL	20496332CRDL	1	09/04/2018	23:32	X	X	X	X	X	X	X	X	X	X	X	X
3028525BLANK	3028525	1	09/04/2018	23:36	X	X	X	X	X	X	X	X	X	X	X	X
PALMER-081318	10443691001	1	09/04/2018	23:39	X		X	X	X	X		X		X		
3038257SD	3038257	5	09/04/2018	23:42	X		X	X	X	X		X		X		
3028527MS	3028527	1	09/04/2018	23:49	X		X	X	X	X		X		X		
3028528MSD	3028528	1	09/04/2018	23:53	X		X	X	X	X		X		X		
3028526LCS	3028526	1	09/05/2018	00:07	X		X	X	X	X		X		X		
20496333CCV	20496333CCV	1	09/05/2018	00:10	X	X	X	X	X	X	X	X	X	X	X	X
20496334CCB	20496334CCB	1	09/05/2018	00:14	X	X	X	X	X	X	X	X	X	X	X	X
RH-06-081418	10443691008	1	09/05/2018	00:17	X		X	X	X	X		X		X		
RH-05-081418	10443691009	1	09/05/2018	00:20			X	X	X	X				X		
RH-47-081418	10443691010	1	09/05/2018	00:24	X		X	X	X	X				X		
RH-47D-081418	10443691011	1	09/05/2018	00:27	X		X	X	X	X				X		
3028529MS	3028529	1	09/05/2018	00:30	X		X	X	X	X				X		
MW-01-081418	10443691007	1	09/05/2018	00:44	X		X	X	X	X		X		X		
20496335CCV	20496335CCV	1	09/05/2018	00:50	X	X	X	X	X	X	X	X	X	X	X	X
20496336CCB	20496336CCB	1	09/05/2018	00:54	X	X	X	X	X	X	X	X	X	X	X	X
20496337CCV	20496337CCV	1	09/05/2018	01:29	X	X	X	X	X	X	X	X	X	X	X	X
20496338CCB	20496338CCB	1	09/05/2018	01:33	X	X	X	X	X	X	X	X	X	X	X	X
20496339CRDL	20496339CRDL	1	09/05/2018	01:36	X	X	X	X	X	X	X	X	X	X	X	X
20496340CCV	20496340CCV	1	09/05/2018	02:14	X	X	X	X	X	X	X	X	X	X	X	X
20496341CCB	20496341CCB	1	09/05/2018	02:17	X	X	X	X	X	X	X	X	X	X	X	X

No sample result should be reported between ICVs or CCVs which do not meet criteria, but before qualifying data based on ICV/CCV recoveries, consult the analysis run log to verify that sample results were reported between the out-of-control calibration standards. Table 6 provides calibration curve correlation requirements, as wells as ICV and CCV percent recovery criteria for differing analyses. Both

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recovery and frequency criteria must be met. If frequency criteria were not met, qualify all affected results as estimated (J). Apply corrective actions in accordance with the rules in Table 7.

Table 6 – Calibration Criteria

Calibration Step	Analysis	Method	Frequency	Control Limits
Calibration Curve Fit	Mercury	SW846 7470 SW846 7470A EPA 245.1	At beginning of run	r ≥ 0.995
ICV			Immediately after instrument calibration and after a continuing calibration failure	90-110% of true value EPA 245.1 - 95-105%
CCV			Every ten samples, and after the last analytical sample	90-110% of true value
Calibration Curve Fit	Metals	SW846 6020 SW846 6020A SW846 6020B EPA 200.8	At beginning of run	r ≥ 0.998
ICV			Immediately after instrument calibration and after a continuing calibration failure	90-110% of true value
CCV			Every ten samples, and after the last analytical sample	90-110% of true value
Calibration Curve Fit	Metals	SW846 6010B SW846 6010C SW846 6010D EPA 200.7	At beginning of run	r ≥ 0.995
ICV			Immediately after instrument calibration and after a continuing calibration failure	90-110% of true value EPA 200.7 - 95-105%
CCV			Every ten samples, and after the last analytical sample	90-110% of true value SW846 6010B - The RSD of the CCV must be < 5%
Calibration Curve Fit	Alkalinity	SM 2320B	At beginning of run	slope 96-106% of true value
pH Calibration Check			Immediately after calibration of pH probe	± 0.10 pH units
ICV			Immediately after instrument calibration and after a continuing calibration failure	90-110% of true value

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Calibration Step	Analysis	Method	Frequency	Control Limits
CCV			Every ten samples, and after the last analytical sample	90-110% of true value
Calibration Curve Fit	NH_3	EPA 350.1	At beginning of run	$r \geq 0.995$
ICV			Immediately after instrument calibration and after a continuing calibration failure	90-110% of true value
CCV			Every ten samples, and after the last analytical sample	90-110% of true value
Calibration Curve Fit	NO_2/NO_3	SM 4500 NO_3-H	At beginning of run	$r \geq 0.995$
ICV			Immediately after instrument calibration and after a continuing calibration failure	90-110% of true value
CCV			Every ten samples, and after the last analytical sample	90-110% of true value
Calibration Curve Fit	TKN	EPA 351.2	At beginning of run	$r \geq 0.995$
ICV			Immediately after instrument calibration and after a continuing calibration failure	90-110% of true value
CCV			Every ten samples, and after the last analytical sample	90-110% of true value
Calibration Curve Fit	DOC/TOC	SM 5310C	At beginning of run	$r \geq 0.995$
ICV			Immediately after instrument calibration and after a continuing calibration failure DOC/TOC analysis by SM 5310C calibration frequency is every six months or as needed. Thus ICV frequency may be six months.	90-110% of true value
High and Low Check Standards			Daily prior to sample analysis unless ICV is run that day.	90-110% of true value

Calibration Step	Analysis	Method	Frequency	Control Limits
CCV			Every ten samples, and after the last analytical sample	90-110% of true value
Calibration Curve Fit	Sulfate	ASTM D516-90	At beginning of run	r ≥ 0.990 Standard at or below RL must recover within 60-140% of true value
ICV			Immediately after instrument calibration and after a continuing calibration failure	80-120% of true value
CCV			Every ten samples, and after the last analytical sample	80-120% of true value
Calibration Curve Fit	Chloride	SM 4500-Cl E	At beginning of run	r ≥ 0.995
ICV			Immediately after instrument calibration and after a continuing calibration failure	90-110% of true value
CCV			Every ten samples, and after the last analytical sample	90-110% of true value
Calibration Curve Fit	Anions (Bromide, chloride, fluoride, sulfate)	EPA 300	At beginning of run	r ≥ 0.990 for each analyte
ICV			Immediately after instrument calibration and after a continuing calibration failure	90-110% of true value
CCV			Every ten samples, and after the last analytical sample	90-110% of true value
Calibration Curve Fit	Fluoride	SM 4500-F-C	At beginning of run	slope 90-110%
ICV			Immediately after instrument calibration and after a continuing calibration failure	90-110% of true value
CCV			Every ten samples, and after the last analytical sample	90-110% of true value
Calibration Curve Fit	Total Phosphorus	SM4500P-E	At beginning of run	r ≥ 0.995

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Calibration Step	Analysis	Method	Frequency	Control Limits
ICV			Immediately after instrument calibration and after a continuing calibration failure	90-110% of true value
CCV			Every ten samples, and after the last analytical sample	90-110% of true value
Calibration Curve Fit	Chemical Oxygen Demand (COD)	SM 5220D EPA 410.4	At beginning of run	$r \geq 0.995$
ICV			Immediately after instrument calibration and after a continuing calibration failure	SM5220D - 95-105% EPA 410.4 - 90-10%
CCV			Every ten samples, and after the last analytical sample	SM5220D - 95-105% EPA 410.4 - 90-10%
Calibration Curve Fit	Orthophosphate-P	SM4500-P B/E	At beginning of run	$r \geq 0.995$
ICV			Immediately after instrument calibration and after a continuing calibration failure	90-110% of true value
CCV			Every ten samples, and after the last analytical sample	90-110% of true value
Calibration Curve Fit	Sulfide	SM4500-S^{2-}D	At beginning of run	$r \geq 0.995$
ICV			Immediately after instrument calibration and after a continuing calibration failure Sulfide analysis by SM4500-S^{2-}D calibration frequency is every six months or as needed. Thus ICV frequency may be six months.	90-110% of true value
High and Low calibration checks			Daily prior to sample analysis unless ICV is run that day.	90-110% of true value
CCV			Every ten samples, and after the last analytical sample	90-110% of true value

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Table 7 - Calibration Action

Calibration Results	Calibration Criteria	Action for Samples	Qualifier Code
Calibration not performed		Qualify all results unusable **(R)**	CQ
Correlation coefficient < the method requirement	See Section 3.4 Tables	Qualify results that are ≥ MDL as estimated **(J)** Qualify non-detects as estimated (UJ)	CR
ICV/CCV %R < 67%	80-120%	Qualify results that are ≥ MDL as estimated low **(J-)** Qualify non-detects as estimated (UJ)	ICV/CCV
ICV/CCV %R < 75%	90-110%		
ICV/CCV %R < 79%	95-105%		
ICV/CCV %R 67-79%	80-120%	Qualify results that are ≥ MDL as estimated low **(J-)** Qualify non-detects as estimated (UJ)	ICV/CCV
ICV/CCV %R 75-89%	90-110%		
ICV/CCV %R 79-104%	95-105%		
ICV/CCV %R 121-	80-120%	Qualify results that are ≥ MDL as estimated high **(J+)** No action for non-detects	ICV/CCV
ICV/CCV %R 111-	90-110%		
ICV/CCV %R 106-119%	95-105%		
ICV/CCV %R > 136%	80-120%	Qualify results that are ≥ MDL as estimated high **(J+)** No action for non-detects	ICV/CCV
ICV/CCV %R > 125%	90-110%		
ICV/CCV %R > 119%	95-105%		
ICV/CCV %R > 175%	80-120%	Qualify results that are ≥ MDL as unusable **(R)** No action for non-detects	ICV/CCV
ICV/CCV %R > 160%	90-110%		
ICV/CCV %R > 153%	95-105%		

3.3.3 Laboratory Blank Data

Ideally, all laboratory blanks including initial calibration blanks (ICBs), continuing calibration blanks (CCBs), method blanks (MBs) or preparation blanks (PBs), should be non-detect (U-flagged by the laboratory) or have a reported value ≤ MDL. MDL values are statistically calculated on no less than an annual basis, and these may change year to year. When referencing laboratory QC requirements in project QAPPs, be aware that validation criteria for blanks differs from laboratory blank criteria. The validator assesses blanks to the MDL; whereas the laboratory blank criteria is a value < the RL or ½ the RL. Were the laboratory to repeat an analysis until all blank results were non-detect, they could consume the entire client sample volume and be unable to perform laboratory QCs such as a matrix spike or duplicate sample.

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All laboratory analyses require a MB (can also be identified as a preparation blank) at a frequency of one MB per batch of 20 of fewer samples. All analyses which require calibrations (calibration samples are not applicable to solids determinations, i.e. TDS and TSS) must analyze an ICB at the beginning of the analytical run, immediately after the ICV, and a CCB every ten samples, immediately after the CCV. Ensure that the required frequency was met when assessing laboratory blank results; and qualify affected data as estimated (J) if the frequency was not met.

Where there are positive and negative blank detections, qualification is assigned based on the highest absolute blank value. Once a data validation qualifier has been applied, add reason code "ICB", "CCB", or "MB" as appropriate. For laboratory ICB/CCB results > than laboratory criteria, analysis should have been terminated, and the contamination source determined and corrected. If necessary, the instrument should have been recalibrated and any sample analyzed since the last in-control blank should have been re-analyzed. When assessing ICB/CCB results, ensure that sample results were reported between out of control blank detections. For method blank detections > criteria, each sample result <10x the blank value should have been re-digested (if applicable) and reanalyzed. If sample results are non-detect, this is not required. If re-analysis was not possible (sample volume was consumed), the sample results should be qualified. To assign qualification to sample results based on a laboratory blank detection, the instrument value must be used. The instrument value is calculated by dividing the sample result by the dilution factor.

BPSOU project data dumps include a field "ModifiedMDL (mg/L)". In the event of laboratory blank detections, the maximum blank detection which affected sample results should be entered in this field. For other projects, check with the project manager or project quality assurance officer prior to entering any laboratory blank results into this field. Although sample results are assessed in comparison to laboratory blank results, a laboratory blank result should never be substituted for the MDL. A laboratory blank result is a single result at a single point in time; whereas MDLs are determined by a statistical process every thirteen months, at a minimum. MDLs are determined by analyzing a minimum of seven spiked samples and seven blank samples in at least three batches on three separate calendar days, with the analyses spread across all instruments to which the MDL will be applied. Statistical analysis is then applied to the sample results to determine the MDL.

Table 8 – Laboratory Blank Action

Lab Blank Result	Sample Results	Action for Samples
≥ MDL, but ≤ 1.5x MDL	Non-detect	No action
	> MDL, but ≤ RL	No action
	> RL	No action
> 1.5x MDL	Non-detect	No action
	≥ MDL, but ≤ RL	Qualify results as estimated non-detect (UJ)
	> RL, but ≤ 10x blank	Qualify results as estimated high (J+)
	>10x blank value	No action
> RL	See action above for 1.5x MDL value. If any sample results are > MDL and < 10x the blank value, note in the Data Validation Summary that the laboratory failed to re-digest (if applicable) and reanalyze the affected samples. Reanalysis is not required for sample results < MDL or > 10X the blank detection.	

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Lab Blank Result	Sample Results	Action for Samples
< 2x (-MDL)	Non-detect	Qualify results as estimated (UJ)
	≤ 5x absolute blank	Qualify results as estimated low (J-)
Lab Sample	**Report Level**	**Frequency[1]**
ICB	L3, L4	At beginning of analytical run, immediately after ICV
CCB	L3, L4	One in every 10 samples, immediately after CCV
MB	L2, L3, L4	One per batch of 20 or fewer samples

[1]ICB/CCB samples not applicable to gravimetric (solids) analyses

3.3.4 Contract Required Detection Limit (CRDL)

Not all analyses include a contract required detection limit (CRDL) sample. These samples simply check the recoveries of standards which have analyte present at the CRDL. Recoveries are calculated by:

$$\%R = \frac{Found\ Value}{True\ Value}\ x\ 100$$

Frequency and recovery requirements for CRDL samples are detailed below in Table 9. Note that these laboratory samples will only be reported in Level 3 or 4 packages. Corrective actions for out of control CRDL samples are presented in Table 10.

Table 9 - Contract Required Detection Limit/Reporting Limit Criteria

Calibration Step[1]	Analysis	Method	Frequency	Control Limits
Contract Required Detection Limit (CRDL)	Mercury	SW846 7470 SW846 7470A EPA 245.1	At the beginning of each run.	70-130%
	Metals	SW846 6020 SW846 6020A SW846 6020B EPA 200.8	At the beginning of each run for every analyte of interest 6020A - at the beginning of each run and at the end of each analytical batch	6020/200.8 - 60-140% 6020A - 70-130% 6020B - 80-120%
	Metals	SW846 6010B EPA 200.7	At the beginning of each run for every analyte of interest	60-140%

[1]Reported only in Level 3 and Level 4 packages

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Table 10 – CRDL Action

Calibration Criteria	Action for Samples	Qualifier Code
CRDL < method criteria	J-	CRQL
CRDL > method criteria	J+	CRQL

3.3.5 Interference Check Sample (ISC) Results

An interference check sample (ICS) is applicable to ICP-MS and ICP-AES analyses, and the purpose is to determine the instrument's capability to overcome common interferences. These samples will be reported only in Level 3 or Level 4 packages. The ICS consists of two solutions, solution A and solution AB. Solution A contains high concentrations of interferents, while solution AB contains the interferents and mid-range concentrations of the target analytes. The two solutions are run consecutively, at the beginning of the analytical sequence, but not before the ICV. The ICSA is run first, followed by the ICSAB, which is immediately followed by a CCV. ICS recovery are calculated by:

$$\%R = \frac{Found\ Value}{True\ Value}\ x\ 100$$

Table 11 states ICS criteria; while corrective actions for out of control results can be found in Table 12. Should data be qualified for out of control ICS recoveries, assign reason code "ICS". Typically, raw data does not contain results for interferents within the samples (this is the concentration of an interferent in each sample, this is not the sample result). If the raw data does contain interferent concentrations for each sample, apply the actions in Table 12 to all samples which have interferent concentrations within 10% of the ICS interferent concentrations. If the raw data does not contain sample interferent concentrations, use Table 12 to assess samples based only on the ICS results.

Table 11 – Interference Check Sample Criteria

Calibration Step[1]	Analysis	Method	Frequency	Control Limits
Interference Check Sample (ICS)	Metals	SW846 6020 SW846 6020A SW846 6020B EPA 200.8	At the beginning of each analytical sequence, or a minimum of twice per 8-hour shift, whichever is more frequent.	80-120% R for analytes included in the ICS, < RL for analytes not included in the ICS
	Metals	SW846 6010B SW846 6010C SW846 6010D EPA 200.7		

[1]Reported only in Level 3 and Level 4 packages

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Table 12 – Interference Check Sample Action

ICS Results	Action for Samples
ICS not analyzed at required frequency	Qualify all results as unusable **(R)**
ICS not analyzed in proper sequence	Use professional judgment
ICS %R <50%	Qualify results ≥ MDL as estimated low (J-) Qualify non-detects as unusable **(R)**
ICS %R 50-79% [or ICS found value is < (true value – 2xRL), whichever is lower]	Qualify results ≥ MDL as estimated low (J-) Qualify non-detects as estimated **(UJ)**
ICS %R >120% [or ICS true value is > (true value + 2xRL), whichever is greater]	Qualify results ≥ MDL as estimated high (J+) No action for non-detects
ICS %R > 150%	Use professional judgement (detects J+ or R) No actions for non-detects
Apply to analyte results ≥ MDLs if samples have detections of analytes not present in ICS. Samples with level of interferents comparable to or higher than interferent levels in the ICS and analyte concentration near the ICS level	Qualify results ≥ MDL as estimated high (J+) No action for non-detects
Apply to negative sample results (but absolute value is ≥ MDL) for analytes that are not present in the ICS solution. Samples with level of interferents comparable to or higher than interferent levels in the ICS	Qualify detects < 10x the | negative result | as estimated low **(J-)** Qualify non-detects as estimated (UJ)

3.3.6 Internal Standards Relative Intensity

Internal standards are applicable to ICP analyses, and these are reported only in Level 3 and Level 4 packages. An internal standard is added to each client sample and the response is monitored throughout the run. The internal standard is made up of analytes which are not typically seen in environmental samples, such as thorium, germanium, scandium, or indium[115], among others. The purpose is to detect instrument drift. The internal standard response is compared to the standard's initial response in the calibration blank. Control limits for internal standards are presented in Table 13, while Table 14 presents actions for out of control responses. Qualified data should be given the validation code "IS".

Table 13 – Internal Standards Relative Response Criteria

Calibration Step[1]	Analysis	Method	Frequency	Control Limits
Internal Standard Response	Metals	SW846 6020 SW846 6020A SW846 6020B EPA 200.8	Monitor signal intensity throughout the analytical run.	**6020** - Any one internal standard's absolute intensity in ICB/CCB and ISCAB must be within 80-120% of original intensity in associated calibration blank, The absolute intensity of any one standard in samples and remaining QC samples must be within 30-120% of original intensity in associated calibration blank. **6020A/6020B** - Response in standards and samples 70-125% of response in associated blank **EPA 200.8** - Response in standards and samples 60-125% of response in associated blank
		SW846 6010B SW846 6010C SW846 6010D EPA 200.7		Response in standards and samples 70-130% of response in associated blank

[1]Reported in Level 3 and Level 4 packages

Table 14 – Internal Standards Response Action

Calibration Criteria	Action for Samples	Qualifier Code
Internal standard response < method criteria and sample was not diluted and re-analyzed	J-	IS
Internal standard response > method criteria and sample was not diluted and re-analyzed	J+	IS

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3.3.7 Laboratory Control Sample (LCS) Result

A laboratory control sample (LCS) is required for nearly all analyses. The LCS is a DI water spiked with known concentrations of all target analytes. For soils analyses, the LCS is a spiked non-metal containing matrix. The LCS may also be referred to as a laboratory fortified blank (LFB). The LCS is assessed on percent recovery:

$$\%R = \frac{Found\ Value}{True\ Value}\ x\ 100$$

If the LCS recovery does not fall within control limits, the analysis should be terminated, the problem corrected, and associated samples reanalyzed. Occasionally, an LCS result is not within criteria, and analysis proceeds. This may occur if all client sample volume has been consumed. Frequency and control limits for the LCS are provided in

Table 15, while Table 16 provides corrective actions.

Several analyses require a laboratory control sample duplicate. This is a separate sample from the laboratory duplicate. The LCSD is a duplicate sample of the LCS. The LCSD percent recovery is assessed identically to the LCS; and in addition, the LCS/LCSD are assessed in terms of relative percent difference (RPD) between the two sample results. This tests the laboratory's repeatability, or precision. The LCS/LCSD RPD is determined by:

$$RPD\% = \frac{|LCS - LCSD|}{\frac{LCS + LCSD}{2}} \times 100$$

Like the LCS, if the LCSD recovery does not fall within the control limits specified in, analysis should be terminated, the problem corrected, and affected samples re-analyzed. If LCS/LCSD precision (RPD) is outside of control limits, analysis should be terminated, the problem corrected, and affected samples re-analyzed. The frequency and control limits for the few samples which required LCSDs are included in

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Table 15, and the corrective action for unacceptable RPDs is at the end of Table 16.

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Table 15 - Laboratory Control Sample/Laboratory Control Sample Duplicate Criteria

Calibration Step[1]	Analysis	Method	Frequency	Control Limits
Laboratory Control Spike (LCS)	Mercury	SW846 7470 SW846 7470A EPA 245.1	One in every 20 samples	80-120% of true value **EPA 245.1** - 85-115% of true value
	Metals	SW846 6020 SW846 6020A SW846 6020B EPA 200.8		80-120% of true value **EPA 200.8** - 85-115% of true value
	Metals	SW846 6010B SW846 6010C SW846 6010D EPA 200.7		80-120% **EPA 200.7** - 85-115% of true value
	Alkalinity (LCS & LCSD)	SM 2320B		90-110% of true value RPD ≤ 20%
	TDS/TSS (LCS & LCSD)[2]	SM 2540C/2540D		80-120% of true value RPD ≤ 10%
	NH_3	EPA 350.1		90-110% of true value
	NO_2/NO_3	SM 4500 NO_3-H		
	TKN	EPA 351.2		
	DOC/TOC	SM 5310C		80-120% of true value RPD ≤ 20%
	Sulfate (LCS & LCSD)	ASTM D516-90		
	Chloride	SM 4500-Cl E		90-110% of true value
	Anions	EPA 300.0		

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Calibration Step[1]	Analysis	Method	Frequency	Control Limits
	Fluoride	SM 4500-F-C		
	Total Phosphorus	SM4500P E		
	COD	SM 5220D EPA 410.4		
	Sulfide	SM4500-S^{2-}D		80-120% of true value
	Orthophosphate-P	SM4500-P B/E		90-110% of true value

[1]Reported in Level 2, Level 3, and Level 4 packages

[2]TDS/TSS – LCSD sample may be analyzed in place of laboratory duplicate at the analyst's discretion. TDS/TSS duplicate sample frequency criteria of 1 in 10 samples must be met.

Table 16 – Laboratory Control Sample/Lab Control Sample Duplicate Action

Calibration Results[1]	Calibration Criteria	Action for Samples	Qualifier Code
LCS not performed at required frequency	1 per batch of 20 or fewer samples	Use professional judgement. Investigate why LCS was not performed. At a minimum, qualify detects as estimated (J) and non-detects as estimated non-detect (UJ)	LCS
LCS %R < 40%	70-130% 80-120% 85-115% 90-110%	Qualify results that are ≥ MDL as estimated low **(J-)** Qualify non-detects rejected (R)	LCS
LCS %R 40-69%	70-130%	Qualify results that are ≥ MDL as estimated low **(J-)** Qualify non-detects as estimated non-detect (UJ)	LCS
LCS %R 40-79%	80-120%		
LCS %R 40-84%	85-115%		
LCS %R 40-89%	90-110%		
LCS %R 131-150%	70-130%		LCS
LCS %R 121-150%	80-120%		

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Calibration Results[1]	Calibration Criteria	Action for Samples	Qualifier Code
LCS %R 116-150%	85-115%	Qualify results that are ≥ MDL as estimated high **(J+)** No action for non-detects	
LCS %R 111-150%	90-110%		
LCS %R >150%	70-130% 80-120% 85-115% 90-110%	Qualify results that are ≥ MDL Rejected **(R)** No action for non-detects	LCS
LCS/LCSD RPD > criteria (10%, 20%)	≤ 10% RPD ≤ 20% RPD	Qualify affected results as estimated (J)	RPD

[1]LCS results are reported in Level 2, Level 3, and Level 4 packages

3.3.8 Laboratory Duplicate Sample (LDS) Results

The purpose of the laboratory duplicate sample is to assess the laboratory and method precision. The LDS is a second aliquot of a client sample that is treated identically to the primary aliquot. Known field blanks should not be used for the LDS. LDS frequency and acceptance criteria are provided in Table 15. In many cases, the matrix spike duplicate (MSD) is used as the LDS. Refer to Section 3.3.9 for a discussion of MSD samples. As Table 17 indicates, the LDS is assessed on the RPD between the primary and duplicate sample. The RPD is determined by:

$$RPD\% = \frac{|S - D|}{\frac{S + D}{2}} \times 100$$

Where S = sample

D = duplicate

Table 18 provides corrective actions for LDS RPDs greater than criteria. The criteria in Table 18 are applicable when both the primary and duplicate sample concentrations are ≥ 5X the RL. If either the primary or duplicate sample result is < 5X the RL, a difference ≤ the RL between the two results is acceptable.

In assessing LDS RPDs, the sample matrix should be considered. If the parent sample used for the laboratory duplicate sample is dissimilar from other samples in the laboratory quality control (QC) batch, then only the parent sample and samples similar to the parent should be qualified. Sample similarity can be assessed by considering sample field data (pH, SC, ORP), site and sampling documentation (sample location, soil moisture, soil type) and laboratory data such as TSS, TDS, alkalinity, reactive sulfide, and anions. The sample data itself can also be used, such as very high analyte concentrations compared to all other samples in the laboratory QC batch. If sample similarity cannot be assessed, or the validator is not confident that samples are dissimilar, the most conservative approach is to qualify all samples in the QC

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batch. Since the sample matrix is considered, any field collected blank which consists of DI water should not be qualified because of LDS RPDs that exceed acceptance criteria. Known field blank samples should not be used as the LDS primary sample; but given that the analyst may not be aware of which samples are blanks, there are times that this occurs. Should LDS RPDs be greater than acceptance criteria, and the parent sample was a blank (DI water), the blank, along with all other samples in the QC batch, would be qualified. Although the blank matrix is dissimilar to the other samples, a duplicate sample of DI water with an RPD > acceptance criteria indicates a problem; thus, all associated data should be qualified.

Greater variability is expected in solid samples (soil) than in aqueous samples; thus, LDS criteria for solid samples is ≤ 35% RPD. For solid sample results < 5X the RL, a difference ≤ 2XRL between the primary and duplicate result is acceptable.

Several analyses require more than one LDS; thus, two LDS samples are analyzed per QC batch. If only one of the RPDs exceeds criteria, qualifications result. Apply qualifications only to the failing analytes. For example, an anion analysis by EPA 300 may include bromide, chloride, fluoride, and sulfate. If only the bromide RPD exceeds criteria, only bromide results would be qualified. Data qualified for LDS RPD is given the reason code "RPD". Note that in many cases, the MSD sample is used for the LDS.

Table 17 – Laboratory Duplicate Sample Criteria

Calibration Step[1]	Analysis	Method	Frequency	Control Limits
Laboratory Duplicate Sample (LDS)	Mercury (MSD)	SW846 7470 SW846 7470A EPA 245.1	One in every 20 samples	≤ 20% RPD
	Metals (MSD)	SW846 6020 SW846 6020A SW846 6020B EPA 200.8	One in every 20 samples	≤ 20% RPD
	Metals (MSD)	SW846 6010B SW846 6010C SW846 6010D EPA 200.7	One in every 20 samples	≤ 20% RPD
	Alkalinity (MSD)	SM2320B	One in every 10 samples	≤ 20% RPD
	NH_3 (MSD)	EPA 350.1	One in every 10 samples	≤ 20% RPD
	NO2/NO3 (MSD)	SM 4500 NO_3-H	One in every 10 samples	≤ 30% RPD
	TKN (MSD or alternate)	EPA 351.2	One in every 20 samples	≤ 20% RPD

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Calibration Step[1]	Analysis	Method	Frequency	Control Limits
	TDS/TSS	SM 2540C/2540D	One in every 10 samples	≤ 10 % RPD
	DOC/TOC	SM 5310C	One in every 20 samples	≤ 25% RPD
	Sulfate (MSD)	ASTM D516-90	One in every 10 samples	≤ 30% RPD
LDS	Chloride (MSD)	SM 4500-Cl E	One in every 10 samples	≤ 20% RPD
	Anions (MSD)	EPA 300.0	One in every 10 samples	≤ 20% RPD
	Fluoride (MSD)	SM 4500-F-C	One in every 10 samples	≤ 20% RPD
	Total Phosphorus (MSD)	SM4500P E	One in every 10 samples	≤ 30% RPD
	COD (MSD or alternate)	SM 5220D EPA 410.4	One in every 10 samples	≤ 20% RPD
	Sulfide	SM4500-S^{2-}D	One in every 10 samples	≤ 20% RPD
	Orthophosphate-P (MSD)	SM4500-P B/E	One in every 10 samples	≤ 30% RPD
LDS	Metals (solids)[2]	Above applicable analyses	One in every 20 samples	≤ 35% RPD

[1]Reported in Level 2, Level 3, and Level 4 packages
[2]Additional Solids see Section XX

Table 18 – Laboratory Duplicate Sample Action

Duplicate Sample Results[1]	Action for Samples
LDS not performed at required frequency	Use professional judgement. Investigate why LDS was not analyzed. At a minimum, qualify detects as estimated (J) and non-detects as estimated (UJ)
Both original and duplicate sample results ≥ 5X RL and RPD > Table 17 criteria	Qualify results that are ≥ MDL as estimated **(J)** Qualify non-detects as estimated non-detect (UJ)

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Duplicate Sample Results[1]	Action for Samples
Original sample or duplicate sample < 5X the RL (including non-detects) and absolute difference between sample and duplicate > RL	Qualify results that are ≥ MDL as estimated **(J)** Qualify non-detects as estimated non-detect (UJ)
Both original and duplicate sample results ≥ 5X RL and RPD < Table 17 criteria	No action
Original sample or duplicate sample < 5X the RL (including non-detects) and absolute difference between sample and duplicate < RL	No action

[1]Reported in Level 2, Level 3, and Level 4 packages

3.3.9 Matrix Spike (MS) Results

The matrix spike (MS) sample is a client sample spiked with a known amount of analyte. The purpose of the MS is to assess the effect of the sample matrix on the preparation and measurement methods. Often, the laboratory duplicate sample requirement is met by analyzing a matrix spike duplicate (MSD) sample. The MSD is a duplicate of the MS, that is, the same parent sample that is used for the MS is used for the MSD. The spike concentration(s) of the MSD is identical to the concentration(s) used for the MS. MS/MSD frequency and acceptance criteria vary with analysis, and these can be found in Table 19. As Table 19 indicates, MS/MSD samples are assessed on recovery and on the RPD between the two samples. RPD assessment was discussed above under laboratory duplicate samples, Section 3.3.8. MS/MSD recoveries are assessed as:

$$\%R = \frac{SSR - SR}{SA} \times 100$$

Where SSR = spiked sample result

SR = sample result

SA = spike added

When the sample result (SR) is < the MDL, use a value of zero for SR when calculating the recovery.

Table 20 provides corrective actions for out of control MS/MSD recoveries. Corrective actions for MS/MSD RPDs are discussed in Section 3.3.8. An exception to the qualification criteria in Table 20 is when the parent sample concentration is ≥ 4X the spike concentration. If this is the case, the recovery criteria are waived.

In assessing MS/MSD recoveries, the sample matrix should be considered. If the parent sample used for the MS/MSD is dissimilar from other samples in the laboratory quality control (QC) batch, then only the parent sample and samples similar to the parent should be qualified. Sample similarity can be assessed by considering sample field data (pH, SC, ORP, soil type for solid samples) and laboratory data such as TSS, TDS, alkalinity, reactive sulfide, and anions. The sample data itself can also be used, such as very high analyte concentrations compared to all other samples in the laboratory QC batch. If sample similarity cannot be assessed, or the validator is not confident that samples are dissimilar, the most conservative

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approach is to qualify all samples in the QC batch. Since the sample matrix is considered, any field collected blank which consists of DI water should not be qualified because of MS/MSD recoveries that do not meet acceptance criteria. Known field blank samples should not be used as the MS parent sample; but given that the analyst may not be aware of which samples are blanks, there are times that this occurs. Should MS/MSD recoveries fall outside of acceptance criteria, and the parent sample was a blank (DI water), the blank, along with all other samples in the QC batch, would be qualified. Although the blank matrix is dissimilar to the other samples, out of control recoveries of spiked DI water indicate a problem; thus, all associated data should be qualified.

Many analyses require a matrix spike sample at a 10% frequency; thus, two MS samples, and possibly two MSDs are analyzed per QC batch. If only one of the spiked sample recoveries does not meet criteria, qualifications result. Apply qualifications only to the failing analytes. For example, an ICP analysis by EPA 200.8 may include arsenic, cadmium, copper, lead, manganese, and zinc. If only the zinc recovery does not meet criteria, only zinc results would be qualified. Data qualified for MS or MSD recoveries is given the reason code "MS".

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Table 19 – Matrix Spike/Matrix Spike Duplicate Criteria

Calibration Step[1]	Analysis	Method	Frequency	Control Limits
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Mercury	SW846 7470 SW846 7470A EPA 245.1	One per batch of 20 samples **EPA 245.1** - 1 per batch & if > 11 samples in a batch, an additional MS is required.	80-120% of true value EPA 245.1 - 70-130% of true value ≤ 20% RPD
	Metals	SW846 6020 SW846 6020A SW846 6020B EPA 200.8	MS One in every 20 samples **EPA 200.8** MS - One in every 10 samples MSD one in every 20 samples	75-125% of true value EPA 200.8 - 70-130% of true value ≤ 20% RPD
	Metals	SW846 6010B SW846 6010C SW846 6010D EPA 200.7	MS One in every 20 samples **EPA 200.7** - 1 MS per 10 samples MSD 1 in 20	75-125% of true value EPA 200.7 - 70-130% of true value ≤ 20% RPD
	Alkalinity	SM2320B	One in every 10 samples	80-120% of true value ≤ 20% RPD
	NH_3 (MSD)	EPA 350.1		90-110% of true value ≤ 20% RPD
	NO_2/NO_3	SM 4500 NO_3-H		80-120% of true value ≤ 30% RPD
	TKN	EPA 351.2	MS 1 in 10 MSD 1 in 20	90-110% of true value ≤ 20% RPD

Calibration Step[1]	Analysis	Method	Frequency	Control Limits
MS	DOC/TOC	SM 5310C	One in every 20 samples No MSD	80-120% of true value
MS/MSD	Sulfate	ASTM D516-90	One in every 10 samples	80-120% of true value ≤ 30% RPD
	Chloride	SM 4500-Cl E		80-120% of true value ≤ 20% RPD
	Anions	EPA 300.0	More frequent of 1 per batch or 1 per 10 samples	90-110% of true value ≤ 20% RPD
	Fluoride	SM 4500-F-C	One in every 10 samples	80-120% of true value ≤ 20% RPD
	Total Phosphorus	SM4500P E		80-120% of true value ≤ 30% RPD
MS/MSD (or alternate duplicate sample)	COD	SM 5220D EPA 410.4		SM 5220D 80-120% EPA 410.4 90-110% ≤ 20% RPD
MS	Sulfide	SM4500-S^{2-}D	One in every 20 samples No MSD	75-125% of true value
MS/MSD	Orthophosphate-P	SM4500-P B/E	One in 10 with a max of 2 MS/MSD pairs per batch of 20	80-120% ≤ 30% RPD

[1]Reported in Level 2, Level 3, and Level 4 packages

Table 20 - Matrix Spike/Matrix Spike Duplicate Recovery Action

MS Sample Results[1]	Calibration Criteria	Action for Samples	Qualifier Code
MS not performed at required frequency	As specified in Table 15	Use professional judgement. Investigate why MS was not performed. At a minimum, qualify detects as estimated (J) and non-detects as estimated (UJ)	MS
MS/MSD %R < 30%	70-130%	Qualify results that are ≥ MDL as estimated low **(J-)** Qualify non-detects rejected (R)	MS
	75-125%		
	80-120%		
	90-110%		
MS/MSD %R 30-69%	70-130%	Qualify results that are ≥ MDL as estimated low **(J-)** Qualify non-detects as estimated (**UJ**)	MS
MS/MSD %R 30-74%	75-125%		
MS/MSD %R 30-79%	80-120%		
MS/MSD %R 30-89%	90-110%		
MS/MSD %R >130%	70-130%	Qualify results that are ≥ MDL as estimated high **(J+)** No action for non-detects	MS
MS/MSD %R > 125%	75-125%		
MS/MSD %R >120%	80-120%		
MS/MSD %R >110%	90-110%		

[1]MS/MSD results are reported in Level 2, Level 3, and Level 4 packages

3.3.9.1 Post digestion spike

Post-digestion spike (PDS) samples are applicable to ICP data and are required for the analytical methods listed in Table 21 when the MS recovery falls outside of criteria, and the parent sample concentration is < 4X the spike concentration. The PDS must be assessed only for the analytes that did not meet MS criteria. A matrix spike sample is spiked at the beginning of the sample preparation process, while the PDS is spiked after the sample has gone through preparation. Table 21 provides PDS criteria, while Table 22 provides corrective actions for samples that do not meet MS and PDS criteria. If a PDS is analyzed, the results are reported only in Level 3 and Level 4 packages. When assessing PDS recoveries consider sample similarity in the manner described in Section 3.3.9 Matrix Spike (MS) Results. Since a PDS sample is analyzed only when there is an MS result which does not meet criteria, the code "MS,PDS" should be used when samples are qualified for MS and PDS results.

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Table 21– Post Digestion Spike Criteria

Laboratory QC[1]	Analysis	Method	Frequency	Control Limits[2]
Post digestion spike	Metals	SW846 6020 SW846 6020A SW846 6020B	One per QC batch if MS/MSD recovery outside of 75-125%	6020/6020A 80-120% of true value 6020B 75-125% of true value
	Metals	SW846 6010B SW846 6010C SW846 6010D	One per QC batch if MS/MSD recovery outside of 75-125%	6010B 85-120% of true value 6010C 80-120% of true value 6010D 75-125% of true value

[1]Reported in Level 3 and Level 4 packages

[2]Post digestion spike assessment is required only for elements failing MS recovery criteria

Table 22 – Post Digestion Spike Action

Spiked Sample Results	Applicable Method	Action for Samples
MS %R < 30% PDS %R < 75%	6020B 6010D	Qualify results that are ≥ MDL as estimated low **(J-)** Qualify non-detects as rejected (R)
MS %R < 30% PDS %R < 80%	6020 6020A 6010C	Qualify results that are ≥ MDL as estimated low **(J-)** Qualify non-detects as rejected (R)
MS %R < 30% PDS %R < 85%	6010B	Qualify results that are ≥ MDL as estimated low **(J-)** Qualify non-detects as rejected (R)
MS %R < 30% PDS %R ≥ 75%	6020B 6010D	Qualify results that are ≥ MDL as estimated **(J)** Qualify non-detects as estimated (UJ)
MS %R < 30% PDS %R ≥ 80%	6020 6020A 6010C	Qualify results that are ≥ MDL as estimated **(J)** Qualify non-detects as estimated (UJ)
MS %R < 30% PDS %R ≥ 85%	6010B	Qualify results that are ≥ MDL as estimated **(J)** Qualify non-detects as estimated (UJ)
MS %R 30-74%	6020B 6010D	Qualify results that are ≥ MDL as estimated low **(J-)** Qualify non-detects as estimated (UJ)

Spiked Sample Results	Applicable Method	Action for Samples
PDS %R < 75%		
MS %R 30-74% PDS %R < 80%	6020 6020A 6010C	Qualify results that are ≥ MDL as estimated low **(J-)** Qualify non-detects as estimated (UJ)
MS %R 30-74% PDS %R < 85%	6010B	Qualify results that are ≥ MDL as estimated low **(J-)** Qualify non-detects as estimated (UJ)
MS %R > 125% PDS %R > 125%	6020B 6010D	Qualify results that are ≥ MDL as estimated high **(J+)** No action for non-detects
MS %R > 125% PDS %R > 120%	6020 6020A 6010B 6010C	Qualify results that are ≥ MDL as estimated high **(J+)** No action for non-detects
MS %R > 125% PDS %R ≤ 125%	6020B 6010D	Qualify results that are ≥ MDL as estimated **(J)** No action for non-detects
MS %R > 125% PDS %R ≤ 120%	6020 6020A 6010B 6010C	Qualify results that are ≥ MDL as estimated **(J)** No action for non-detects
MS %R < 30% No PDS performed	All	Qualify results that are ≥ MDL as estimated low **(J-)** Qualify non-detects as rejected (R)
MS %R 30-74% No PDS performed	All	Qualify results that are ≥ MDL as estimated low **(J-)** Qualify non-detects as estimated (UJ)
MS %R 75-125% No PDS is required	All	No action
MS %R > 125% No PDS performed	All	Qualify results that are ≥ MDL as estimated high **(J+)** No action for non-detects

[1]Reported in Level 3 and Level 4 packages

[2]Post digestion spike assessment is required only for elements failing MS recovery criteria

3.3.10 Serial Dilution Sample Results

Serial dilution (SD) samples are applicable to ICP data. The SD is a client sample which is diluted by a factor of five. The dilution corrected result (SD result x 5) should be within a specific percent difference of the original sample result, for samples in which the original concentration is sufficiently high. The SD sample is an indication of physical or chemical interferences within the sample matrix. Serial dilution % difference is determined by:

$$\%Difference = \frac{|I - S|}{I} \times 100$$

Where I = initial sample result

S = serial dilution result

Since SD samples assess matrix interference, field blank samples should not be used for the initial sample. Additionally, as with MS and PDS samples, sample similarity should be considered when assessing SD results; and since field blank samples are of a differing matrix, they are not qualified for SD samples which do not meet criteria. Serial dilution results are reported only in Level 3 and Level 4 packages. Table 23 specifies analyses for which SD samples are applicable, and acceptance criteria; while Table 24 presents actions for non-compliant SD results. The code "SD" should be used for samples qualified for serial dilution results which do not meet criteria.

Table 23 – Serial Dilution Criteria

Laboratory QC[1]	Analysis	Method	Frequency	Control Limits
Serial Dilution (SD)	Metals	SW846 6020 SW846 6020A SW846 6020B EPA 200.8	One in every batch of 20 or fewer samples	6020/6020A/EPA 200.8 - 1:5 dilution 10% difference of original result when original sample is ≥ 50X the MDL 6020B - 20% difference of 1:5 dilution of MS or samples with concentration 25X the lower limit of quantification in parent sample
Serial Dilution (SD)	Metals	SW846 6010B SW846 6010C SW846 6010D EPA 200.7	One in every batch of 20 or fewer samples	6010B, 6010C - 10% difference of original result when original sample is > 10X the RL. 6010D - 20% difference of 1:5 dilution of MS or samples with concentration 25X the lower limit of quantification in parent sample EPA 200.7 - 10% difference for original samples ≥ 50X the MDL

[1]Reported in Level 3 and Level 4 packages

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Table 24 – Serial Dilution Action

SD Result[1]	Applicable Method	Original Sample Concentration	Action for Samples	Qualifier Code
SD not performed at required frequency	One per batch of 20 or fewer samples	NA	Use professional judgement. Investigate why MS was not performed. At a minimum, qualify detects as estimated (J) and non-detects as estimated (UJ)	SD
SD %Difference (%D) > 10%	SW846 6020 SW846 6020A EPS 200.8 SW846 6010B SW846 6010C EPA 200.7	≥ 50X MDL	Qualify results that are ≥ MDL as estimated **(J)** Qualify non-detects as estimated non-detect (UJ)	SD
SD %D > 10%	SW8466020 SW846 6020A EPA 200.8 SW846 6010B SW846 6010C EPA 200.7	< 50X MDL	No Qualification	
SD %D > 20%	SW846 6020B SW846 6010D	> 25X lower limit of quantification or 1:5 dilution of MS	Qualify results that are ≥ MDL as estimated (J) Qualify non-detects as estimated non-detect (UJ)	SD
SD %D > 20%	SW846 6020B SW846 6010D	< 25X lower limit of quantification	No Qualification	

[1]Serial dilution results are reported in Level 3 and Level 4 packages

3.4 Data Validation Process for Analytical Parameters

This section outlines validation steps by analytical parameter (mercury, metals, alkalinity, etc.). Since laboratory packages present calibration and QC results by parameter, the most efficient way to assess data is by parameter, rather than by QC sample. That is, assess all mercury calibration and QC sample results, next assess all ICP calibration and QC sample results, and so on, rather than assessing blank results for all analyses, assess LCS results for all analyses, etcetera. In assessing laboratory data for each parameter, use the limits and corrective actions provided in Section 3.3.

3.4.1 Mercury Assessment

Mercury analyses are assessed for the calibration and QC samples found in Table 25.

3.4.2 Metals Assessment

Metals analyses are assessed for the calibration and QC samples found in Table 26.

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3.4.3 Alkalinity Assessment

Alkalinity analyses are assessed for the calibration and QC samples found in Table 27

3.4.4 Solids (TDS/TSS) Assessment

Nitrate + nitrite analyses are assessed for the calibration and QC samples found in

ED_014362_00001316-00057

Table 28.

3.4.1 Nitrate +Nitrite Assessment

Nitrate + nitrite analyses are assessed for the calibration and QC samples found in Table 29

3.4.2 Ammonia Assessment

Ammonia analyses are assessed for the calibration and QC samples found in Table 30.

3.4.3 Total Kjeldahl Nitrogen (TKN) Assessment

TKN analyses are assessed for the calibration and QC samples found in Table 31.

3.4.4 Dissolved/Total Organic Carbon (DOC/TOC) Assessment

DOC and TOC analyses are assessed for the calibration and QC samples found in

ED_014362_00001316-00058

Table 32. The analytical method for DOC and TOC is identical; the difference is that a sample to be analyzed for DOC is field filtered.

3.4.5 Sulfate Analysis by ASTMD 516-90 Assessment

Sulfate analyses by ASTMD 516-90 are assessed for the calibration and QC samples found in Table 33.

3.4.6 Total Phosphorus Assessment

Total phosphorus analyses are assessed for the calibration and QC samples found in

ED_014362_00001316-00059

Table 34

3.4.7 Chloride Analysis by SM4500-Cl E Assessment

Chloride analyses by SM4500-CL E are assessed for the calibration and QC samples found in

ED_014362_00001316-00060

Table 35.

3.4.8 Anion Analysis (Bromide, Chloride, Fluoride, and Sulfate) by EPA 300 Assessment

Anion analyses (bromide, chloride, fluoride, and sulfate) by EPA Method 300 are assessed for the calibration and QC samples found in

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Table 36.

3.4.9 Fluoride Assessment

Fluoride analyses by SM4500-F-C analyses are assessed for the calibration and QC samples found in Table 37.

3.4.10 Sulfide Assessment

Sulfide analyses are assessed for the calibration and QC samples found in

ED_014362_00001316-00062

Table 38.

3.4.11 Chemical Oxygen Demand (COD) Assessment

Chemical oxygen demand (COD) analyses are assessed for the calibration and QC samples found in Table 39

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3.4.12 Orthophosphate Assessment

Orthophosphate analyses are assessed for the calibration and QC samples found in Table 40.

ED_014362_00001316-00064

Table 25 - Mercury Calibration and Laboratory QC Sample Requirements

Laboratory Calibration/ QC Sample	Method	Frequency	Control Limits	Action Table	Level 2 Report	Level 3 Report	Level 4 Report
Calibration Curve Fit	SW846 7470 SW846 7470A EPA 245.1	Once in 24 hours or each time the instrument is set up	r ≥ 0.995	Table 7	No	Yes	Yes
ICV	SW846 7470 SW846 7470A EPA 245.1	Immediately after instrument calibration and after a continuing calibration failure	90-110% of true value EPA 245.1 - 95-105%R	Table 7	No	Yes	Yes
CCV	SW846 7470 SW846 7470A EPA 245.1	1 in 10 samples, and after the last analytical sample	90-110% of true value	Table 7	No	Yes	Yes
CRDL	SW846 7470 SW846 7470A EPA 245.1	At the beginning of each run.	SW846 7470/7470A 80-120%R 245.1 70-130%R	Table 9	No	Yes	Yes
ICB	SW846 7470 SW846 7470A EPA 245.1	Immediately after instrument calibration, following the CCV	< MDL	Table 8	No	Yes	Yes
CCB	SW846 7470 SW846 7470A EPA 245.1	1 in 10 samples and after the last analytical sample, following CCVs	< MDL	Table 8	No	Yes	Yes
MB	SW846 7470 SW846 7470A EPA 245.1	1 per batch of ≤ 20 samples	< MDL	Table 8	Yes	Yes	Yes

Laboratory Calibration/ QC Sample	Method	Frequency	Control Limits	Action Table	Level 2 Report	Level 3 Report	Level 4 Report
LCS	SW846 7470 SW846 7470A EPA 245.1	1 per batch of ≤ 20 samples	80-120% R EPA 245.1 - 85-115%R	Table 16	Yes	Yes	Yes
LDS (MSD suffices as LDS)	SW846 7470 SW846 7470A EPA 245.1	1 per batch of ≤ 20 samples	≤ 20% RPD	Table 18	Yes	Yes	Yes
MS/MSD	SW846 7470 SW846 7470A EPA 245.1	1 per batch of ≤ 20 Samples EPA 245.1 - 1 per batch & if > 11 samples in a batch, an additional MS is required.	80-120%R EPA 245.1 - 70-130%R ≤ 20% RPD	Table 20	Yes	Yes	Yes

Table 26 – Metals Calibration and Laboratory QC Sample Requirements

Laboratory QC	Method	Frequency[1]	Control Limits[1]	Action Table	Level 2 Report	Level 3 Report	Level 4 Report
Tuning	SW846 6020 SW846 6020A SW846 6020B EPA 200.8	Prior to calibration Tune solution analyzed five times, consecutively	mass calibration within 0.1 amu % RSD of aboslute signals < 5%	Table 5	No	Yes	Yes
Calibration Curve Fit	SW846 6020 SW846 6020A SW846 6020B EPA 200.8	Once in 24 hours or each time the instrument is set up	r ≥ 0.998	Table 7	No	Yes	Yes

Laboratory QC	Method	Frequency[1]	Control Limits[1]	Action Table	Level 2 Report	Level 3 Report	Level 4 Report
Calibration Curve Fit	SW846 6010B SW846 6010C SW846 6010D EPA 200.7	Once in 24 hours or each time the instrument is set up	$r \geq 0.995$	Table 7	No	Yes	Yes
ICV	SW846 6020 SW846 6020A SW846 6020B EPA 200.8	Immediately after instrument calibration and after a continuing calibration failure	90-110% of true value	Table 7	No	Yes	Yes
ICV	SW846 6010B SW846 6010C SW846 6010D EPA 200.7	Immediately after instrument calibration and after a continuing calibration failure	90-110% R EPA 200.7 - 95-105%R	Table 7	No	Yes	Yes
CCV	SW846 6020 SW846 6020A SW846 6020B EPA 200.8	1 in every 10 samples, and after the last analytical sample	90-110% R	Table 7	No	Yes	Yes
CCV	SW846 6010B SW846 6010C SW846 6010D EPA 200.7	1 in every 10 samples, and after the last analytical sample	90-110% R SW846 6010B - The RSD of the CCV must be < 5%	Table 7	No	Yes	Yes
CRDL	SW846 6020 SW846 6020A SW846 6020B EPA 200.8	At the beginning of each run for every analyte of interest	6020/200.8 - 60-140% R 6020A - 70-130% R 6020B - 80-120% R	Table 10	No	Yes	Yes
CRDL	SW846 6010B EPA 200.7	At the beginning of each run for every analyte of interest	60-140% R	Table 10	No	Yes	Yes

Laboratory QC	Method	Frequency[1]	Control Limits[1]	Action Table	Level 2 Report	Level 3 Report	Level 4 Report
ICB	SW846 6020 SW846 6020A SW846 6020B EPA 200.8	At beginning of analytical run, immediately after CCV	< MDL	Table 8	No	Yes	Yes
ICB	SW846 6010B SW846 6010C SW846 6010D EPA 200.7	At beginning of analytical run, immediately after ICV	< MDL	Table 8	No	Yes	Yes
CCB	SW846 6020 SW846 6020A SW846 6020B EPA 200.8	1 in every 10 samples, immediately after CCV	< MDL	Table 8	No	Yes	Yes
CCB	SW846 6010B SW846 6010C SW846 6010D EPA 200.7	1 in every 10 samples, immediately after CCV	< MDL	Table 8	No	Yes	Yes
MB	SW846 6020 SW846 6020A SW846 6020B EPA 200.8	1 in every 20 samples	< MDL	Table 8	Yes	Yes	Yes
MB	SW846 6010B SW846 6010C SW846 6010D EPA 200.7	1 in every 20 samples	< MDL	Table 8	Yes	Yes	Yes
ICS	SW846 6020 SW846 6020A SW846 6020B EPA 200.8	At the beginning of each analytical sequence, or a minimum of twice per 8-hour shift, whichever is more frequent.	80-120% R for analytes included in the ICS, < RL for analytes not included in the ICS	Table 12	No	Yes	Yes

Laboratory QC	Method	Frequency[1]	Control Limits[1]	Action Table	Level 2 Report	Level 3 Report	Level 4 Report
ICS	SW846 6010B SW846 6010C SW846 6010D EPA 200.7	At the beginning of each analytical sequence, or a minimum of twice per 8-hour shift, whichever is more frequent.	80-120% R for analytes included in the ICS, < RL for analytes not included in the ICS	Table 12	No	Yes	Yes
Internal Standard Response	SW846 6020 SW846 6020A SW846 6020B EPA 200.8	Monitor signal intensity throughout the analytical run.	6020 - absolute intensity in ICB/CCB and ISCAB 80-120% of original intensity in associated calibration blank, absolute intensity in samples and remaining QC samples 30-120% of original intensity in associated calibration blank. 6020A/6020B - 70-125% EPA 200.8 - 60-125%	Table 14			
Internal Standard Response	SW846 6010B SW846 6010C SW846 6010D EPA 200.7	Monitor signal intensity throughout the analytical run.	70-130% R	Table 14	No	Yes	Yes
LCS	SW846 6020 SW846 6020A SW846 6020B EPA 200.8	1 in every 20 samples	80-120% R EPA 200.8 - 85-115% R	Table 16	Yes	Yes	Yes
LCS	SW846 6010B SW846 6010C SW846 6010D EPA 200.7	1 in every 20 samples	80-120% R EPA 200.7 - 85-115% R	Table 16	Yes	Yes	Yes

Laboratory QC	Method	Frequency[1]	Control Limits[1]	Action Table	Level 2 Report	Level 3 Report	Level 4 Report
LDS	SW846 6020 SW846 6020A SW846 6020B EPA 200.8	1 in every 20 samples MSD suffices as LDS	20% RPD	Table 18	Yes	Yes	Yes
LDS	SW846 6010B SW846 6010C SW846 6010D EPA 200.7	1 in every 20 samples MSD suffices as LDS	20% RPD	Table 18	Yes	Yes	Yes
MS/MSD	SW846 6020 SW846 6020A SW846 6020B EPA 200.8	MS One in every 20 samples EPA 200.8 MS - One in every 10 samples MSD one in every 20 samples	25-125% R EPA 200.8 - 70-130% R ≤ 20% RPD	Table 20	Yes	Yes	Yes
MS/MSD	SW846 6010B SW846 6010C SW846 6010D EPA 200.7	MS One in every 20 samples EPA 200.7 - 1 MS per 10 samples MSD 1 in 20	75-125% R EPA 200.7 - 70-130% R 20% RPD	Table 20	Yes	Yes	Yes
PDS	SW846 6020 SW846 6020A SW846 6020B	1 per QC batch if MS/MSD recovery outside of 75-125%	6020/6020A 80-120% R 6020B 75-125% of true value	Table 22	No	Yes	Yes
PDS	SW846 6010B SW846 6010C SW846 6010D	1 per QC batch if MS/MSD recovery outside of 75-125%	6010B 85-120% R 6010C 80-120% R 6010D 75-125% R	Table 22	No	Yes	Yes
SD	SW846 6020 SW846 6020A SW846 6020B EPA 200.8	1 in every 20 samples	6020/6020A/EPA 200.8 - 1:5 dilution 10% D of original result when original sample is ≥ 50X the MDL 6020B - 20% D of 1:5 dilution of MS or samples with	Table 24	No	Yes	Yes

Laboratory QC	Method	Frequency[1]	Control Limits[1]	Action Table	Level 2 Report	Level 3 Report	Level 4 Report
			concentration 25X the lower limit of quantification in parent sample				
SD	SW846 6010B SW846 6010C SW846 6010D EPA 200.7	1 in every 20 samples	6010B, 6010C - 10% D of original result when original sample is > 10X the RL. 6010D - 20% D of 1:5 dilution of MS or samples with concentration 25X the lower limit of quantification in parent sample EPA 200.7 - 10% D for original samples ≥ 50X the MDL	Table 24	No	Yes	Yes

Table 27 – Alkalinity Calibration and Laboratory QC Requirements

Laboratory QC	Method	Frequency	Control Limits	Action Table	Level 2 Report	Level 3 Report	Level 4 Report
Calibration Curve Fit	SM 2320B	At the beginning of run	slope 96-106% of true value	Table 7	No	Yes	Yes
pH Calibration Check	SM 2320B	Immediately after pH probe calibration	± 0.10 pH units	Table 7	No	Yes	Yes

Laboratory QC	Method	Frequency	Control Limits	Action Table	Level 2 Report	Level 3 Report	Level 4 Report
ICV	SM 2320B	Immediately after instrument calibration and after a continuing calibration failure	90-110% R	Table 7	No	Yes	Yes
CCV	SM 2320B	1 in every ten samples, and after the last analytical sample	90-110% R	Table 7	No	Yes	Yes
ICB	SM 2320B	Immediately after instrument calibration, following the ICV	< MDL	Table 8	No	Yes	Yes
CCB	SM 2320B	One in every 10 samples	< MDL	Table 8	No	Yes	Yes
MB	SM 2320B	1 in every 20 samples	< MDL	Table 8	Yes	Yes	Yes
LCS	SM 2320B	1 in every 20 samples	90-110% R	Table 16	Yes	Yes	Yes
LCSD	SM 2320B	1 in every 20 samples	20% RPD	Table 16	Yes	Yes	Yes
LDS	SM 2320B	1 in every 10 samples MSD suffices for LDS	20% RPD	Table 18	Yes	Yes	Yes
MS/MSD	SM2320B	One in every 10 samples	80-120% R 20% RPD	Table 20	Yes	Yes	Yes

Table 28 – TDS/TSS Laboratory QC Requirements

Laboratory QC	Method	Frequency	Control Limits	Action Table	Level 2 Report	Level 3 Report	Level 4 Report
MB	SM 2540C/D	1 in every 20 samples	< MDL	Table 8	Yes	Yes	Yes
LCS	SM 2540C/D	1 in every 20 samples	80-120% R	Table 16	Yes	Yes	Yes
LCSD	SM 2540C/D	1 in every 20 samples (if analyzed)	10% RPD	Table 16	Yes	Yes	Yes
LDS	SM 2540C/D	1 in every 10 samples	10% RPD	Table 18	Yes	Yes	Yes

Table 29 – Nitrate + Nitrite Calibration and Laboratory QC Requirements

Laboratory QC	Method	Frequency	Control Limits	Action Table	Level 2 Report	Level 3 Report	Level 4 Report
Calibration Curve Fit	SM 4500 NO_3-H	At the beginning of run	$r \geq 0.995$	Table 7	No	Yes	Yes
ICV	SM 4500 NO_3-H	Immediately after instrument calibration and after a continuing calibration failure	90-110% R	Table 7	No	Yes	Yes
CCV	SM 4500 NO_3-H	One in every ten samples, and after the last analytical sample	90-110% R	Table 7	No	Yes	Yes
ICB	SM 4500 NO_3-H	Immediately after instrument calibration, immediately following ICV	< MDL	Table 8	No	Yes	Yes
CCB	SM 4500 NO_3-H	1 in every 10 samples	< MDL	Table 8	No	Yes	Yes
MB	SM 4500 NO_3-H	1 in every 20 samples	< MDL	Table 8	Yes	Yes	Yes
LCS	SM 4500 NO_3-H	1 in every 20 samples	90-110% R	Table 16	Yes	Yes	Yes

Laboratory QC	Method	Frequency	Control Limits	Action Table	Level 2 Report	Level 3 Report	Level 4 Report
LDS	SM 4500 NO_3-H	1 in every 20 samples MSD suffices as LDS	≤ 30% RPD	Table 18	Yes	Yes	Yes
MS/MSD	SM 4500 NO_3-H	1 in every 20 samples	80-120% R ≤ 30% RPD	Table 20	Yes	Yes	Yes

Table 30 – Ammonia Calibration and Laboratory QC Sample Requirements

Laboratory QC	Method	Frequency	Control Limits	Action Table	Level 2 Report	Level 3 Report	Level 4 Report
Calibration Curve Fit	EPA 350.1	At the beginning of run	$r \geq 0.995$	Table 7	No	Yes	Yes
ICV	EPA 350.1	Immediately after instrument calibration and after a continuing calibration failure	90-110% R	Table 7	No	Yes	Yes
CCV	EPA 350.1	1 in every ten samples, and after the last analytical sample	90-110% R	Table 7	No	Yes	Yes
ICB	EPA 350.1	Immediately after instrument calibration, immediately following ICV	< MDL	Table 8	No	Yes	Yes
CCB	EPA 350.1	1 in every ten samples, and after the last analytical sample	< MDL	Table 8	No	Yes	Yes
MB	EPA 350.1	1 in every 20 samples	< MDL	Table 8	Yes	Yes	Yes
LCS	EPA 350.1	1 in every 20 samples	90-110% R	Table 16	Yes	Yes	Yes
LDS	EPA 350.1	1 in every 10 samples MSD suffices as LDS	≤ 20% RPD	Table 18	Yes	Yes	Yes
MS/MSD	EPA 350.1	1 in every 20 samples	90-110% R 20% RPD	Table 20	Yes	Yes	Yes

Table 31 – TKN Calibration and Laboratory QC Samples Requirements

Laboratory QC	Method	Frequency	Control Limits	Action Table	Level 2 Report	Level 3 Report	Level 4 Report
Calibration Curve Fit	EPA 351.121	At the beginning of run	$r \geq 0.995$	Table 7	No	Yes	Yes
ICV	EPA 351.2	Immediately after instrument calibration and after a continuing calibration failure	90-110% R	Table 7	No	Yes	Yes
CCV	EPA 351.2	1 in every 10 samples, and after the last analytical sample	90-110% R	Table 7	No	Yes	Yes
ICB	EPA 351.2	At beginning of analytical run, immediately after CCV	< MDL	Table 8	No	Yes	Yes
CCB	EPA 351.2	1 in every 10 samples, and after the last analytical sample	< MDL	Table 8	No	Yes	Yes
MB	EPA 351.2	1 in every 20 samples	< MDL	Table 8	Yes	Yes	Yes
LCS	EPA 351.2	1 in every 20 samples	90-110% R	Table 16	Yes	Yes	Yes
LDS	EPA 351.2	1 in every 20 samples (MSD or alternate)	≤ 20% RPD	Table 18	Yes	Yes	Yes
MS/MSD	EPA 351.2	MS 1 in every 10 samples MSD 1 in 20	90-110% R	Table 20	Yes	Yes	Yes

Table 32 – DOC/TOC Calibration and Laboratory QC Samples Requirements

Laboratory QC	Method	Frequency	Control Limits	Action Table	Level 2 Report	Level 3 Report	Level 4 Report
Calibration Curve Fit	SM 5310C	At the beginning of run	$r \geq 0.995$	Table 7	No	Yes	Yes
ICV	SM 5310C	Immediately after instrument calibration and after a continuing calibration failure DOC/TOC analysis by SM 5310C calibration frequency is every six months or as needed. Thus, ICV frequency may be six months.	90-110% R	Table 7	No	Yes	Yes
High and Low Standard Check	SM 5310C	Daily prior to sample analysis unless ICV is run that day		Table 7			
CCV	SM5310 C	One in every ten samples, and after the last analytical sample	90-110% R	Table 7	No	Yes	Yes
ICB	SM5310 C	At beginning of analytical run, immediately after CCV	< MDL	Table 8	No	Yes	Yes
CCB	SM5310 C	1 in every 10 samples, and after the last analytical sample	< MDL	Table 8	No	Yes	Yes
MB	SM5310 C	1 in every 20 samples	< MDL	Table 8	Yes	Yes	Yes
LCS	SM5310 C	1 in every 20 samples	80-120% R	Table 16	Yes	Yes	Yes
LDS	SM5310 C	1 in every 20 samples	≤ 25% RPD	Table 18	Yes	Yes	Yes
MS	SM5310 C	MS 1 in every 20 samples	80-120% R	Table 20	Yes	Yes	Yes

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Table 33 – Sulfate by ASTM D516-90 Calibration and Laboratory QC Sample Requirements

Laboratory QC	Method	Frequency	Control Limits	Action Table	Level 2 Report	Level 3 Report	Level 4 Report
Calibration Curve Fit	ASTM D516-90	At the beginning of run	$r \geq 0.990$	Table 7	No	Yes	Yes
ICV	ASTM D516-90	Immediately after instrument calibration and after a continuing calibration failure	80-120% R	Table 7	No	Yes	Yes
CCV	ASTM D516-90	1 in every 10 samples, and after the last analytical sample	80-120% R	Table 7	No	Yes	Yes
ICB	ASTM D516-90	At beginning of analytical run, immediately after CCV	< MDL	Table 8	No	Yes	Yes
CCB	ASTM D516-90	1 in every 10 samples, and after the last analytical sample	< MDL	Table 8	No	Yes	Yes
MB	ASTM D516-90	1 in every 20 samples	< MDL	Table 8	Yes	Yes	Yes
LCS	ASTM D516-90	1 in every 20 samples	80-120% R	Table 16	Yes	Yes	Yes
LCSD	ASTM D516-90	1 in every 20 samples	≤ 20% RPD	Table 16	Yes	Yes	Yes
LDS	ASTM D516-90	1 in every 10 samples MSD suffices as LDS	≤ 30% RPD	Table 18	Yes	Yes	Yes
MS/MSD	ASTM D516-90	1 in every 10 samples	80-120% R 30% RPD	Table 20	Yes	Yes	Yes

Table 34 - Total Phosphate by SM4500P-E Calibration and Laboratory QC Sample Requirements

Laboratory QC	Method	Frequency	Control Limits	Action Table	Level 2 Report	Level 3 Report	Level 4 Report
Calibration Curve Fit	SM4500P-E	At the beginning of run	$r \geq 0.995$	Table 7	No	Yes	Yes
ICV	SM4500P-E	Immediately after instrument calibration and after a continuing calibration failure	90-110% R	Table 7	No	Yes	Yes
CCV	SM4500P E	1 in every 10 samples, and after the last analytical sample	90-110% R	Table 7	No	Yes	Yes
ICB	SM4500P E	At beginning of analytical run, immediately after CCV	< MDL	Table 8	No	Yes	Yes
CCB	SM4500P E	1 in every 10 samples, and after the last analytical sample	< MDL	Table 8	No	Yes	Yes
MB	SM4500P E	One in every 20 samples	< MDL	Table 8	Yes	Yes	Yes
LCS	SM4500P E	One in every 20 samples	80-120% R	Table 16	Yes	Yes	Yes
LDS	SM4500P E	One in every 20 samples MSD suffices as LDS	20% RPD	Table 18	Yes	Yes	Yes
MS/MSD	SM4500P E	One in every 20 samples	80-120% R	Table 20	Yes	Yes	Yes

Table 35 – Chloride by SM4500-Cl E Calibration and Laboratory QC Sample Requirements

Laboratory QC	Method	Frequency	Control Limits	Action Table	Level 2 Report	Level 3 Report	Level 4 Report
Calibration Curve Fit	SM 4500-Cl E	At the beginning of run	$r \geq 0.995$	Table 7	No	Yes	Yes
ICV	SM 4500-Cl E	Immediately after instrument calibration and after a continuing calibration failure	90-110% R	Table 7	No	Yes	Yes
CCV	SM 4500-Cl E	1 in every 10 samples, and after the last analytical sample	90-110% R	Table 7	No	Yes	Yes
ICB	SM 4500-Cl E	At beginning of analytical run, immediately after CCV	< 1/2 RL	Table 8	No	Yes	Yes
CCB	SM 4500-Cl E	1 in every 10 samples, and after the last analytical sample	< 1/2 RL	Table 8	No	Yes	Yes
MB	SM 4500-Cl E	1 in every 20 samples	< 1/2 RL	Table 8	Yes	Yes	Yes
LCS	SM 4500-Cl E	1 in every 20 samples	90-110% R	Table 16	Yes	Yes	Yes
LDS	SM 4500-Cl E	1 in every 10 samples MSD suffices as LDS	≤ 20% RPD	Table 18	Yes	Yes	Yes
MS/MSD	SM 4500-Cl E	1 in every 10 samples	80-120% R	Table 20	Yes	Yes	Yes

Table 36 – Anion (Bromide, chloride, fluoride, sulfate) Analysis by EPA 300.0 Calibration and Laboratory QC Sample Requirements

Laboratory QC	Method	Frequency	Control Limits	Action Table	Level 2 Report	Level 3 Report	Level 4 Report
Calibration Curve Fit	EPA 300.0	At the beginning of run	$r \geq 0.990$	Table 7	No	Yes	Yes
ICV	EPA 300.0	Immediately after instrument calibration and after a continuing calibration failure	90-110% R	Table 7	No	Yes	Yes
CCV	EPA 300.0	1 in every 10 samples, and after the last analytical sample	90-110% R	Table 7	No	Yes	Yes
ICB	EPA 300.0	At beginning of analytical run, immediately after CCV	< MDL	Table 8	No	Yes	Yes
CCB	EPA 300.0	1 in every 10 samples, and after the last analytical sample	< MDL	Table 8	No	Yes	Yes
MB	EPA 300.0	1 in every 20 samples	< MDL	Table 8	Yes	Yes	Yes
LCS	EPA 300.0	1 in every 20 samples	90-110% R	Table 16	Yes	Yes	Yes
LDS	EPA 300.0	1 in every 10 samples MSD suffices as LDS	≤ 20% RPD	Table 18	Yes	Yes	Yes
MS/MSD	EPA 300.0	1 in every 10 samples	90-110% R	Table 20	Yes	Yes	Yes

Table 37 - Fluoride Analysis by SM4500-F-C Calibration and Laboratory QC Sample Requirements

Laboratory QC	Method	Frequency	Control Limits	Action Table	Level 2 Report	Level 3 Report	Level 4 Report
Calibration Curve	SM 4500-F-C	Daily, prior to analysis	Slope=90-110%	Table 7	No	Yes	Yes
ICV	SM 4500-F-C	Immediately after instrument calibration and after a continuing calibration failure Sulfide analysis by SM4500-S^{2-}D calibration frequency is every six months or as needed. Thus, ICV frequency may be six months.	90-110% R	Table 7	No	Yes	Yes
CCV	SM 4500-F-C	1 in every 10 samples, and after the last analytical sample	90-110% R	Table 7	No	Yes	Yes
ICB	SM 4500-F-C	At beginning of analytical run, immediately after CCV	< MDL	Table 8	No	Yes	Yes
CCB	SM 4500-F-C	1 in every 10 samples, and after the last analytical sample	< MDL	Table 8	No	Yes	Yes
MB	SM 4500-F-C	One in every 20 samples	< MDL	Table 8	Yes	Yes	Yes
LCS	SM 4500-F-C	One in every 20 samples	90-110% R	Table 16	Yes	Yes	Yes
LDS	SM 4500-F-C	One in every 10 samples	20% RPD	Table 18	Yes	Yes	Yes
MS/MSD	SM 4500-F-C	One in every 10 samples	80-120% R	Table 20	Yes	Yes	Yes

Table 38 - Sulfide Analysis by SM4500-S^2-D Calibration and Laboratory QC Sample Requirements

Laboratory QC	Method	Frequency	Control Limits	Action Table	Level 2 Report	Level 3 Report	Level 4 Report
Calibration Curve Fit	SM4500-S^{2-}D	At the beginning of run	$r \geq 0.995$	Table 7	No	Yes	Yes
ICV	SM4500-S^{2-}D	Immediately after instrument calibration and after a continuing calibration failure Sulfide analysis by SM4500-S^{2-}D calibration frequency is every six months or as needed. Thus ICV frequency may be six months.	90-110% R	Table 7	No	Yes	Yes
High calibration check	SM4500-S^{2-}D	Daily prior to sample analysis unless ICV is run that day.	90-110% R	Table 7	No	Yes	Yes
Low calibration check	SM4500-S^{2-}D	Daily prior to sample analysis unless ICV is run that day.	90-110% R	Table 7	No	Yes	Yes
CCV	SM4500-S^{2-}D	One in every ten samples, and after the last analytical sample	90-110% R	Table 7	No	Yes	Yes
ICB	SM4500-S^{2-}D	At beginning of analytical run, immediately after CCV	< MDL	Table 8	No	Yes	Yes
CCB	SM4500-S^{2-}D	1 in every 10 samples, and after the last analytical sample	90-110% R	Table 8	No	Yes	Yes
MB	SM4500-S^{2-}D	One in every 20 samples	< MDL	Table 8	Yes	Yes	Yes
LCS	SM4500-S^{2-}D	One in every 20 samples	80-120% R	Table 16	Yes	Yes	Yes
LDS	SM4500-S^{2-}D	One in every 20 samples	20% RPD	Table 18	Yes	Yes	Yes
MS	SM4500-S^{2-}D	One in every 20 samples	75-125% of true value	Table 20	Yes	Yes	Yes

Table 39 – COD Analysis by SM5220D and EPA 410.4 Calibration and Laboratory QC Sample Requirements

Laboratory QC	Method	Frequency	Control Limits	Action Table	Level 2 Report	Level 3 Report	Level 4 Report
Calibration Curve Fit	SM 5220D EPA 410.4	At the beginning of run	$r \geq 0.995$	Table 7	No	Yes	Yes
ICV	SM 5220D EPA 410.4	Immediately after instrument calibration and after a continuing calibration failure	SM5220D - 95-105% EPA 410.4 - 90-10%	Table 7	No	Yes	Yes
CCV	SM 5220D EPA 410.4	One in every ten samples, and after the last analytical sample	SM5220D - 95-105% EPA 410.4 - 90-10%	Table 7	No	Yes	Yes
ICB	SM 5220D EPA 410.4	At beginning of analytical run, immediately after CCV	< MDL	Table 8	No	Yes	Yes
CCB	SM 5220D EPA 410.4	1 in every 10 samples, and after the last analytical sample	< MDL	Table 8	No	Yes	Yes
MB	SM 5220D EPA 410.4	One in every 20 samples	< MDL	Table 8	Yes	Yes	Yes
LCS	SM 5220D EPA 410.4	One in every 20 samples	90-110% R	Table 16	Yes	Yes	Yes
LDS	SM 5220D EPA 410.4	One in every 10 samples MSD suffices as LDS	20% RPD	Table 18	Yes	Yes	Yes
MS	SM 5220D EPA 410.4	One in every 10 samples	SM 5220D 80-120% EPA 410.4 90-110%	Table 20	Yes	Yes	Yes

Table 40 – Ortho Phosphate Analysis by SM4500P-G Calibration and Laboratory QC Sample Requirements

Laboratory QC	Method	Frequency	Control Limits	Action Table	Level 2 Report	Level 3 Report	Level 4 Report
Calibration Curve Fit	SM4500P-G	At the beginning of run	$r \geq 0.995$	Table 7	No	Yes	Yes
ICV	SM4500P-G	Immediately after instrument calibration and after a continuing calibration failure	90-110% R	Table 7	No	Yes	Yes
CCV	SM4500P-G	1 in every 10 samples, and after the last analytical sample	90-110% R	Table 7	No	Yes	Yes
ICB	SM4500P-G	At beginning of analytical run, immediately after CCV	< MDL	Table 8	No	Yes	Yes
CCB	SM4500P-G	1 in every 10 samples, and after the last analytical sample	< MDL	Table 8	No	Yes	Yes
MB	SM4500P-G	One in every 20 samples	< MDL	Table 8	Yes	Yes	Yes
LCS	SM4500P-G	One in every 20 samples	90-110% R	Table 16	Yes	Yes	Yes
LDS	SM4500P-G	One in every 10 samples MSD suffices as LDS	30% RPD	Table 18	Yes	Yes	Yes
MS/MSD	SM4500P-G	One in every 10 samples	80-120% R	Table 20	Yes	Yes	Yes

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3.5 Reported Results Authentication

Compare at least 10% of the reported results to the raw data results. This will be possible only with Level 4 reports. When comparing results, correct for sample volumes, dilution factors, and units. The results should be confirmed randomly (i.e. do not pick the first 10% of results reported.) If there is a discrepancy, the laboratory needs to be notified and to submit a report revision with correct results included.

One of the initial steps in Level 4 data validation is confirming that all laboratory calibration and QC samples, as well as all client samples, appear in the raw data. Reported concentrations can be checked when confirming calibration and sample presence in the raw data. Figure 5 below shows the reported results for a client sample which has been assigned laboratory ID 10455965003. Figure 6 displays the Analysis Run Log. From the log, the analysis time and dilution factor can be determined. Note that sample -003 was run at 1X, 10X, and 100X dilutions and different parameters were reported from each of these analyses. Figure 7 shows the sample -003 raw data for the 1X dilution; while Figure 8 shows the raw data for the 10X and 100X dilutions. Note that in the examples below, results from the raw data are in ppb, while the reported values are in ppm. When corrected for units and dilutions, the raw data agrees with the reported values.

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Figure 5 – Reported Results for Client Sample with Laboratory ID 10455965003

ANALYTICAL RESULTS

Project: Rocker
Pace Project No.: 10455965

Sample: [redacted] **Lab ID: 10455965003** Collected: 11/15/18 10:26 Received: 11/17/18 10:00 Matrix: Water

Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
200.8 MET ICPMS, Dissolved	Analytical Method: EPA 200.8 Preparation Method: EPA 200.8								
Arsenic, Dissolved	**0.73**	mg/L	0.00050	0.00011	1	11/28/18 08:59	12/06/18 23:22	7440-38-2	
Cadmium, Dissolved	**0.0011**	mg/L	0.000080	0.000027	1	11/28/18 08:59	12/06/18 23:22	7440-43-9	
Calcium, Dissolved	**88.1**	mg/L	0.40	0.14	10	11/28/18 08:59	12/06/18 23:25	7440-70-2	
Copper, Dissolved	**0.0011**	mg/L	0.0010	0.00022	1	11/28/18 08:59	12/06/18 23:22	7440-50-8	
Iron, Dissolved	**0.26**	mg/L	0.050	0.0054	1	11/28/18 08:59	12/06/18 23:22	7439-89-6	
Lead, Dissolved	**0.000067J**	mg/L	0.00010	0.000039	1	11/28/18 08:59	12/06/18 23:22	7439-92-1	
Magnesium, Dissolved	**22.7**	mg/L	0.010	0.0050	1	11/28/18 08:59	12/06/18 23:22	7439-95-4	
Manganese, Dissolved	**27.8**	mg/L	0.050	0.024	100	11/28/18 08:59	12/06/18 23:28	7439-96-5	
Potassium, Dissolved	**4.8**	mg/L	0.10	0.018	1	11/28/18 08:59	12/06/18 23:22	7440-09-7	
Silicon, Dissolved	**19.5**	mg/L	0.50	0.16	10	11/28/18 08:59	12/06/18 23:25	7440-21-3	
Sodium, Dissolved	**29.3**	mg/L	0.050	0.018	1	11/28/18 08:59	12/06/18 23:22	7440-23-5	
Zinc, Dissolved	**0.0033J**	mg/L	0.0050	0.0019	1	11/28/18 08:59	12/06/18 23:22	7440-66-6	
2320B Alkalinity	Analytical Method: SM 2320B								
Alkalinity, Hydroxide (CaCO3)	ND	mg/L	5.0	1.0	1		11/28/18 08:18		
Alkalinity, Total as CaCO3	**226**	mg/L	5.0	1.0	1		11/28/18 08:18		
Alkalinity,Bicarbonate (CaCO3)	**226**	mg/L	5.0	1.0	1		11/28/18 08:18		
Alkalinity,Carbonate (CaCO3)	ND	mg/L	5.0	1.0	1		11/28/18 08:18		
2540C Total Dissolved Solids	Analytical Method: SM 2540C								
Total Dissolved Solids	**514**	mg/L	20.0	10.0	1		11/21/18 10:57		
ASTM D516 Sulfate Water	Analytical Method: ASTM D516								
Sulfate	**182**	mg/L	25.0	12.0	10		11/30/18 12:31	14808-79-8	
SM4500CI-E Chloride	Analytical Method: SM 4500-Cl E								
Chloride	**28.1**	mg/L	2.0	0.59	1		11/27/18 11:32	16887-00-6	

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Figure 6 – Analysis Run Log Showing Sample 10455965003. Log provides analysis time and dilution factors.

FORM XIII INORGANIC-1
ANALYSIS RUN LOG

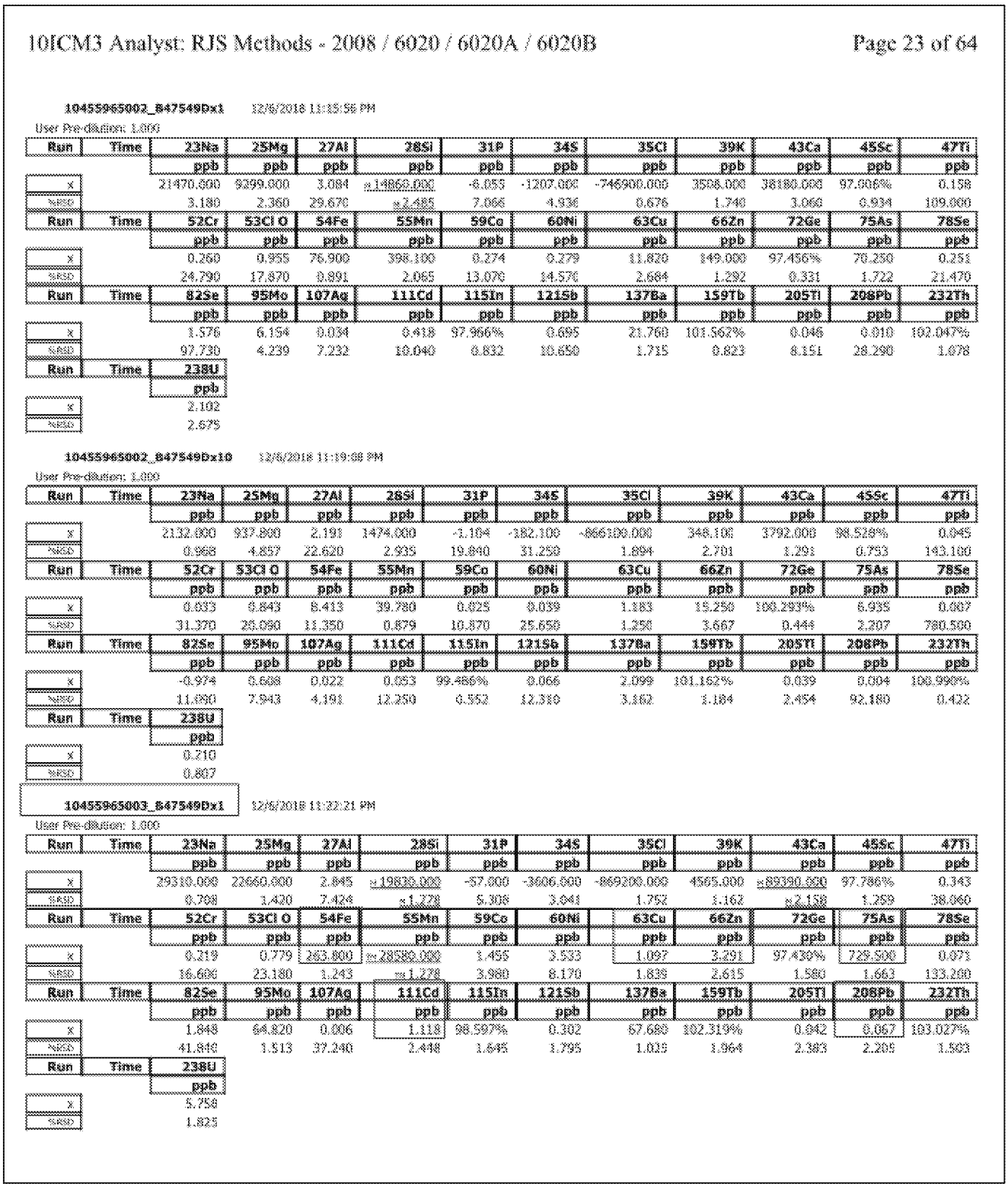
Lab: Pace Analytical - Minnesota SDG No. : 10455965 Contract: Rocker

Instrument: 10ICM3 Analysis: EPA 200.8

Start: 12/06/2018 21:42 End Date: 12/07/2018 02:12

Sample Name	Lab Sample ID	D/F	Date	Time	As	Ca	Cd	Cu	Fe	K	Mg	Mn	Na	Pb	Si	Zn
21235421CAL0	21235421CAL0	1	12/06/2018	21:42	X	X	X	X	X	X	X	X	X	X	X	X
21235422CAL1	21235422CAL1	1	12/06/2018	21:45	X	X	X	X	X	X	X	X	X	X	X	X
21235423CAL4	21235423CAL4	1	12/06/2018	21:48	X	X	X	X	X	X	X	X	X	X	X	X
21235424CAL5	21235424CAL5	1	12/06/2018	21:52	X	X	X	X	X	X	X	X	X	X	X	X
21235425CAL6	21235425CAL6	1	12/06/2018	21:55	X	X	X	X	X	X	X	X	X	X	X	X
21235426CAL2	21235426CAL2	1	12/06/2018	21:58	X	X	X	X	X	X	X	X	X	X	X	X
21235427CAL3	21235427CAL3	1	12/06/2018	22:02	X	X	X	X	X	X	X	X	X	X	X	X
21235428ICV	21235428ICV	1	12/06/2018	22:05	X	X	X	X	X	X	X	X	X	X	X	X
21235429ICB	21235429ICB	1	12/06/2018	22:12	X	X	X	X	X	X	X	X	X	X	X	X
21235430CRDL	21235430CRDL	1	12/06/2018	22:15	X	X	X	X	X	X	X	X	X	X	X	X
21235431ICSA	21235431ICSA	1	12/06/2018	22:19	X	X	X	X	X	X	X	X	X	X	X	X
21235432ICSAB	21235432ICSAB	1	12/06/2018	22:22	X	X	X	X	X	X	X	X	X	X	X	X
21235433CCV	21235433CCV	1	12/06/2018	22:25	X	X	X	X	X	X	X	X	X	X	X	X
21235434CCB	21235434CCB	1	12/06/2018	22:32	X	X	X	X	X	X	X	X	X	X	X	X
3127538BLANK	3127538	1	12/06/2018	22:35	X	X	X	X	X	X	X	X	X	X	X	X
3127539LCS	3127539	1	12/06/2018	22:38	X	X	X	X	X	X	X	X	X	X	X	X
[redacted]	10455965001	1	12/06/2018	22:42	X	X	X	X	X	X	X	X	X	X		X
3139123SD	3139123	5	12/06/2018	22:45	X	X	X	X	X	X	X	X	X	X		X
3127540MS	3127540	1	12/06/2018	22:52	X	X	X	X	X	X	X	X	X	X	X	X
3127541MSD	3127541	1	12/06/2018	22:55	X	X	X	X	X	X	X	X	X	X	X	X
[redacted]	10455965001	10	12/06/2018	22:59											X	
3139123SD	3139123	50	12/06/2018	23:06											X	
21235435CCV	21235435CCV	1	12/06/2018	23:09	X	X	X	X	X	X	X	X	X	X	X	X
21235436CCB	21235436CCB	1	12/06/2018	23:12	X	X	X	X	X	X	X	X	X	X	X	X
[redacted]	10455965002	1	12/06/2018	23:15	X	X	X	X	X	X	X	X	X	X		X
	10455965002	10	12/06/2018	23:19											X	
	10455965003	1	12/06/2018	23:22	X		X	X	X	X	X		X	X		X
	10455965003	10	12/06/2018	23:25		X									X	
	10455965003	100	12/06/2018	23:28								X				
	10455965008	1	12/06/2018	23:32			X	X	X	X	X	X	X	X		X
	10455965008	10	12/06/2018	23:35	X	X									X	

Figure 7 – ICP Raw Data Showing Sample 10455965002 Run at a 1X Dilution



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10455965002_847549Dx1 12/6/2018 11:15:56 PM

User Pre-dilution: 1.000

Run	Time	23Na	25Mg	27Al	28Si	31P	34S	35Cl	39K	43Ca	45Sc	47Ti
		ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb
x		21470.000	9299.000	3.084	≈14860.000	-8.055	-1207.000	-746900.000	3508.000	38180.000	97.006%	0.158
%RSD		3.180	2.360	29.670	≈2.485	7.066	4.936	0.676	1.740	3.060	0.934	109.000

Run	Time	52Cr	53Cl O	54Fe	55Mn	59Co	60Ni	63Cu	66Zn	72Ge	75As	78Se
		ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb
x		0.260	0.955	76.900	398.100	0.274	0.279	11.820	149.000	97.456%	76.250	0.251
%RSD		24.790	17.870	0.891	2.065	13.070	14.570	2.684	1.292	0.331	1.722	21.470

Run	Time	82Se	95Mo	107Ag	111Cd	115In	121Sb	137Ba	159Tb	205Tl	208Pb	232Th
		ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb
x		1.576	6.154	0.034	0.418	97.966%	0.695	21.760	101.562%	0.046	0.010	102.047%
%RSD		97.730	4.239	7.232	10.040	0.832	10.650	1.715	0.823	8.151	28.290	1.078

Run	Time	238U
		ppb
x		2.102
%RSD		2.675

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User Pre-dilution: 1.000

Run	Time	23Na	25Mg	27Al	28Si	31P	34S	35Cl	39K	43Ca	45Sc	47Ti
		ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb
x		2132.000	937.800	2.191	1474.000	-1.104	-382.100	-866100.000	348.106	3792.000	98.528%	0.045
%RSD		0.968	4.857	22.620	2.935	19.840	31.250	1.894	2.701	1.291	0.753	143.100

Run	Time	52Cr	53Cl O	54Fe	55Mn	59Co	60Ni	63Cu	66Zn	72Ge	75As	78Se
		ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb
x		0.033	0.843	8.413	39.780	0.025	0.039	1.183	15.250	100.293%	6.935	0.007
%RSD		31.370	20.090	11.350	0.879	10.870	25.650	1.250	3.667	0.444	2.207	780.500

Run	Time	82Se	95Mo	107Ag	111Cd	115In	121Sb	137Ba	159Tb	205Tl	208Pb	232Th
		ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb
x		-0.974	0.688	0.022	0.053	99.486%	0.066	2.099	101.162%	0.039	0.004	100.990%
%RSD		11.490	7.943	4.191	12.250	0.552	12.310	3.162	1.184	2.454	92.180	0.422

Run	Time	238U
		ppb
x		0.210
%RSD		0.807

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User Pre-dilution: 1.000

Run	Time	23Na	25Mg	27Al	28Si	31P	34S	35Cl	39K	43Ca	45Sc	47Ti
		ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb
x		29310.000	22660.000	2.845	≈19830.000	-57.000	-3606.000	-869200.000	4565.000	≈88390.000	97.786%	0.343
%RSD		0.708	1.420	7.424	≈1.278	6.308	3.041	1.752	1.162	≈2.158	1.269	38.060

Run	Time	52Cr	53Cl O	54Fe	55Mn	59Co	60Ni	63Cu	66Zn	72Ge	75As	78Se
		ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb
x		0.219	0.779	263.800	≈28580.000	1.455	3.533	1.097	3.291	97.430%	729.500	0.071
%RSD		16.606	23.180	1.243	≈1.278	3.980	8.170	1.839	2.615	1.580	1.663	133.260

Run	Time	82Se	95Mo	107Ag	111Cd	115In	121Sb	137Ba	159Tb	205Tl	208Pb	232Th
		ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb
x		1.848	64.820	0.006	1.118	98.597%	0.302	67.680	102.319%	0.042	0.067	103.027%
%RSD		41.846	1.513	37.240	2.448	1.645	1.795	1.035	1.964	2.383	2.209	1.503

Run	Time	238U
		ppb
x		5.756
%RSD		1.825

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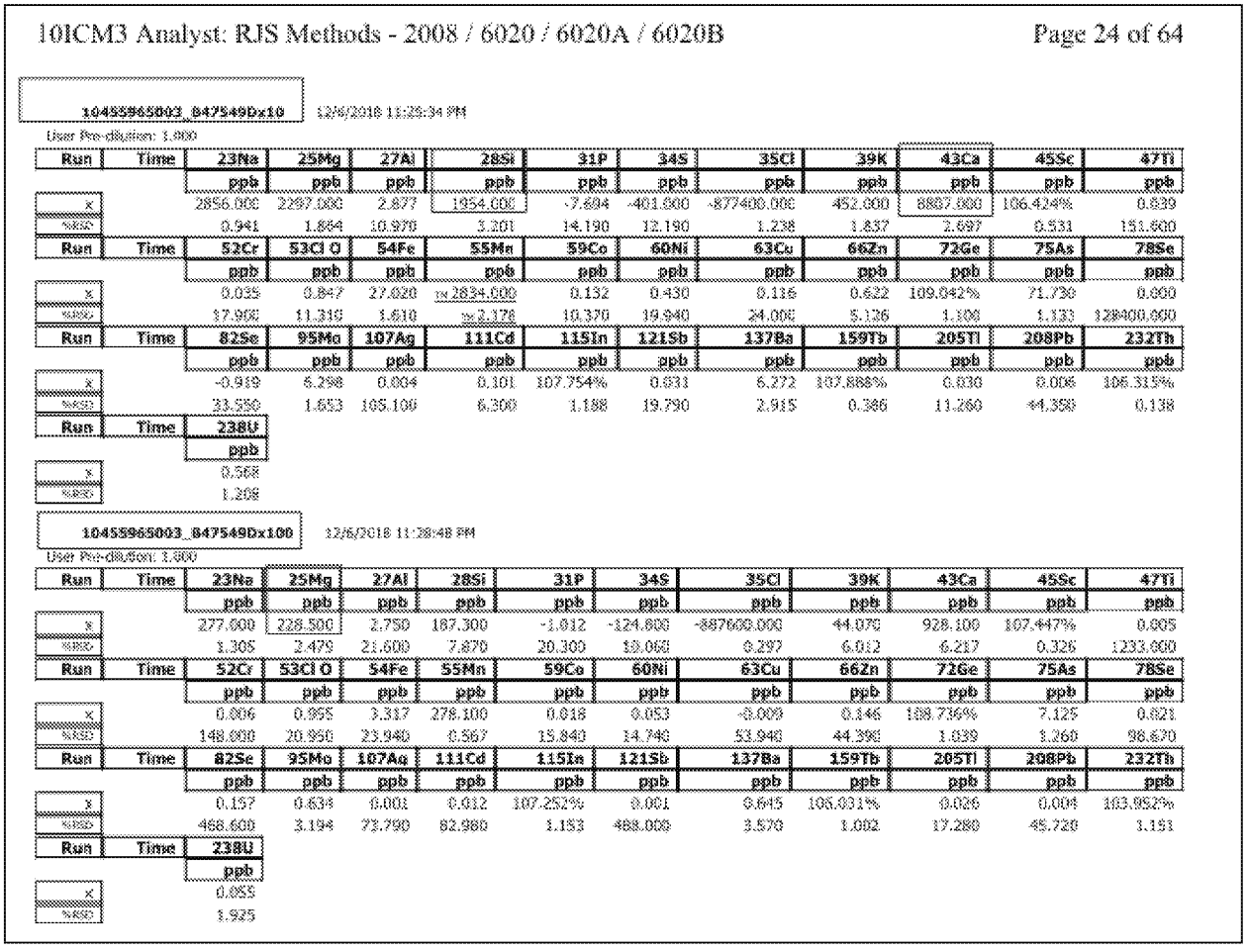
Figure 8 – Raw Data Showing Sample 10455965002 Run at a 10X and 100X Dilutions



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10455965003_847549Dx10 12/6/2018 11:28:34 PM

User Pre-dilution: 1.000

Run	Time	23Na	25Mg	27Al	28Si	31P	34S	35Cl	39K	43Ca	45Sc	47Ti
		ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb
x		2856.000	2297.000	2.877	1954.000	-7.604	-401.900	-877400.000	452.000	8887.000	106.424%	0.839
%RSD		0.941	1.864	10.970	3.201	14.190	12.190	1.238	1.857	2.697	0.531	151.600
Run	Time	52Cr	53Cl O	54Fe	55Mn	59Co	60Ni	63Cu	66Zn	72Ge	75As	78Se
		ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb
x		0.035	0.847	27.020	>> 2834.000	0.132	0.430	0.116	0.622	109.042%	71.730	0.000
%RSD		17.900	11.310	1.610	>> 2.178	10.370	19.940	24.000	5.126	1.308	1.133	128400.000
Run	Time	82Se	95Mo	107Ag	111Cd	115In	121Sb	137Ba	159Tb	205Tl	208Pb	232Th
		ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb
x		-0.919	6.298	0.004	0.101	107.754%	0.033	6.272	107.888%	0.030	0.008	106.315%
%RSD		33.550	1.653	105.100	6.300	1.188	19.790	2.915	0.386	11.260	44.350	0.138
Run	Time	238U										
		ppb										
x		0.568										
%RSD		1.208										

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User Pre-dilution: 1.000

Run	Time	23Na	25Mg	27Al	28Si	31P	34S	35Cl	39K	43Ca	45Sc	47Ti
		ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb
x		277.000	228.500	2.750	187.300	-1.012	-124.800	-887600.000	44.070	928.100	107.447%	0.005
%RSD		1.305	2.479	21.600	7.870	20.300	19.060	0.297	6.012	6.217	0.326	1233.000
Run	Time	52Cr	53Cl O	54Fe	55Mn	59Co	60Ni	63Cu	66Zn	72Ge	75As	78Se
		ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb
x		0.006	0.955	3.317	278.100	0.018	0.053	-0.009	0.146	108.736%	7.125	0.021
%RSD		148.000	20.950	23.940	0.567	15.840	14.746	53.948	44.396	1.039	1.260	98.670
Run	Time	82Se	95Mo	107Ag	111Cd	115In	121Sb	137Ba	159Tb	205Tl	208Pb	232Th
		ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb	ppb
x		0.157	0.634	0.001	0.012	107.252%	0.001	0.645	106.031%	0.026	0.004	103.952%
%RSD		468.600	3.194	73.790	82.980	1.183	488.000	3.570	1.002	17.280	45.720	1.151
Run	Time	238U										
		ppb										
x		0.055										
%RSD		1.925										

3.6 Data Validation Notes to Remember

In addition to the general guidance and method specific guidance discussed above, the validator should keep the items discussed in Section 3.6 in mind.

3.6.1 Laboratory Qualifiers

Sample results come with laboratory qualifiers. The Laboratory Information Management System (LIMS)) automatically qualifies unrounded results lower than the MDL, "U" or "non-detect". Laboratory QC anomalies are also automatically qualified, although the results may meet QC criteria. A common example is a qualifier being applied to the parent sample used for the MS. Very often, the parent sample concentration is greater than 4X the spike concentration; therefore, recovery criteria are waived. Although the LIMS system has applied a qualifier, the validator will not necessarily apply a qualifier. Just like the case narrative, use the laboratory qualifier flags as a tool; do not rely on these flags, as they may be inconsistent with guidance used to assign validation qualifiers.

3.6.2 Laboratory Control Limits

The LIMS may use control limits which differ from those specified in this SOP, or the project QAPP, and control limits may differ among labs. There may be instances when data are flagged by the laboratory,

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and these data will not warrant a data validation qualifier. When assessing data, with the exception of laboratory blanks, the control limits within project QAPPS are the most pertinent reference. The limits tabulated in project QAPPs are the limits laboratories are required to meet; and generally, these limits align with method limits. Aside from laboratory blank criteria, the limits within this SOP also align with method limits. Laboratory blanks are assessed to the MDL during validation, but it is not reasonable to require laboratories to achieve blank results < the MDL.

3.6.3 Multiple Data Qualifiers and DV Reason Codes

In the data assessment process, a data point may be qualified for more than one QC deficiency. For example, a sample result less than the MB result may receive a U qualification. This same data point may be qualified because the laboratory duplicate precision was greater than the acceptable RPD, warranting a J qualification. Each data point is assigned only one qualifier, so an overall qualifier would be applied (see Table 41).

Unlike qualifiers, multiple reason codes may be applied, and these are listed in Table 42 below. If multiple reason codes are used, always list these codes in alphabetical order. For example, if a data point is qualified for matrix spike recovery (MS), laboratory duplicate precision (RPD), and a field blank detection (FB), the codes should be listed as "FB,MS,RPD". All analytical results, data validation qualifiers, and reason codes are stored in a database. A database recognizes "FB,MS,RPD" and "MS,FB,RPD" as two different reason codes. Multiple reason codes must be separated by commas, without any spaces in the text string. Ideally, only one and no more than two reason codes are applied. It is permissible to use more than one reason code; but use discretion in applying codes. For example, if an associated laboratory blank has a detection 10 parts per trillion (ppt) above the MDL and a field blank has a detection 10X the MDL, the FB detection would override the MB detection and only a FB code would be applied.

Table 41 – Multiple Data Qualifiers

Multiple Qualifiers	Overall Qualifier
Data point qualified (U) and either (J), (J+), or (J-)	Qualify result as estimated non-detect (UJ)
Data point qualified a combination of (J) and (J+) or (J) and (J-)	Qualify result as estimated high (J+) or estimated low (J-), respectively
Data point qualified as (J+) and (J-)	Qualify result as estimated (J)
Data point qualified (R) and any other qualifier	Qualify result as rejected (R)

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Table 42 – Data Validation Reason Codes

Code[1]	Definition
A	Laboratory is not accredited for associated analyses
AB	Did not meet level A/B criteria
CC	Correlation coefficient less than 0.995 for instrument calibration
CCB	Continuing calibration blank contamination
CCV	Continuing calibration verification outside limits
CQ	No calibration performed
CRQL	Contract required quantitation limit (CRDL) standard recovery outside quality control limits
DNR	Do not report. An alternate, acceptable result is available.
EB	Equipment blank contamination
ECR	Reported concentration exceeds instrument calibration range
FB	Field blank contamination
FD	Field duplicate RPD outside limits
HT	Holding time exceeded
ICB	Initial calibration blank contamination
ICS	Interference check standard recovery outside limits
ICV	Initial calibration verification outside limits
IP	Incorrect sample preservation
IS	Internal standard recovery outside limits
LCS	Lab control spike recovery is outside quality control limits
MB	Method blank contamination
MDL	Non-detect at MDL value
MI	Matrix interference with analyte quantitation
MS	Matrix spike recovery is outside quality control limits
PDS	Post digestion spike recovery is outside control limits
RB	Equipment rinse blank contamination
RPD	Duplicate sample relative percent difference exceeds QC limits
SD	ICP serial dilution percent difference outside QC limits
SUR	Surrogate recovery is outside QC limits
TB	Trip blank contamination
TIC	Compound was tentatively identified by GC/MS search
[1]Always list reason codes in alphabetical order	

4.0 FIELD DATA VALIDATION

Field data validation includes an assessment of QC samples collected by the sampling team, and a review of sampling documentation and record keeping. Field QC sample assessment is discussed first. Refer to the project QAPP to determine the type and frequency of field QC samples. Some projects require only field duplicates, others require field duplicates and field blanks, and more than one type of field blank may be required. Generally, field QC sample frequency is one field QC sample or sample set (duplicate

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and blank), per 20 primary samples; however, there may be projects which require one field QC set per day. The project QAPP will provide this information.

Data for the majority of TREC projects is managed by the in-house data management team. The team has developed macro-enabled spreadsheets specifically for data validation (referred to as Data Validation Spreadsheet-DVS- below). Spreadsheet format may differ slightly across projects. In the examples within this section, spreadsheets developed for BPSOU are used in examples.

4.1 Data Summary Table Setup

Data summary set-up tables have been developed for BPSOU projects, and these can be found in the project specific folders within the data validation folder. Within each project folder, open the template Project-QC_template (..\2019\SWBF\SWBF-QC_template.xlsx). Figure 9 provides a visual display of the file location for groundwater data validation. These templates check precision of field blanks and results of field blanks. They also include a worksheet on which laboratory QC sample results can be tracked. Data can be copied directly from the DVS into the templates. Within the QC template, the Blank worksheet checks for field blank results which do not meet criteria, and if criteria are not met (≤ 1.5X the MDL), the blank result is multiplied by 10. The worksheet titled Dup calculates the RPD for duplicate pairs. The Dup worksheet also checks sample and duplicate results projectThis file has three worksheets, Lab, Blank, and Dup. A summary of laboratory analyses and any laboratory calibration or QC sample which did not meet criteria will be tracked in the Lab worksheet. All field blank samples will be listed in the Blank worksheet and all field duplicate samples will be listed in the DUP worksheet. When copying data into the worksheets, do not paste over columns containing formulas (yellow shaded).

To find field QC samples within the DVS, filter the Sample Type column for field QC samples. In the location column, any field collected blank will be identified as SWQC or GWQC, and field duplicates will be have a suffix of "-D" (FG-D, IS-D, or AS-D). The primary sample for each duplicate will have suffix of "-N" as in "FG-N") The equations in the QC template check for field blank detections and duplicate sample sets with RPDs > 20%. In the ensuing discussion, this worksheet will be referred to as the Field QC Summary file (FQC).

The templates are set up to include analytical data specific to BPSOU projects; however, this can be revised for other projects.

When pasting field QC data into the FQC, once data is pasted into the Blank worksheet, it needs no further manipulation. The worksheet can be used to check for blank detections > 1.5X the MDL. If a blank detection > 1.5X the MDL occurs, the spreadsheet calculates 10X the blank detection. Data pasted into the Dup worksheet must be manipulated to pair sample and duplicate results. Once data is pasted into the worksheet, it should be sorted by sample number, then by parameter and duplicate analysis, and duplicate pairs should be paired side by side. The column headings can be used to guide the copy/paste process.

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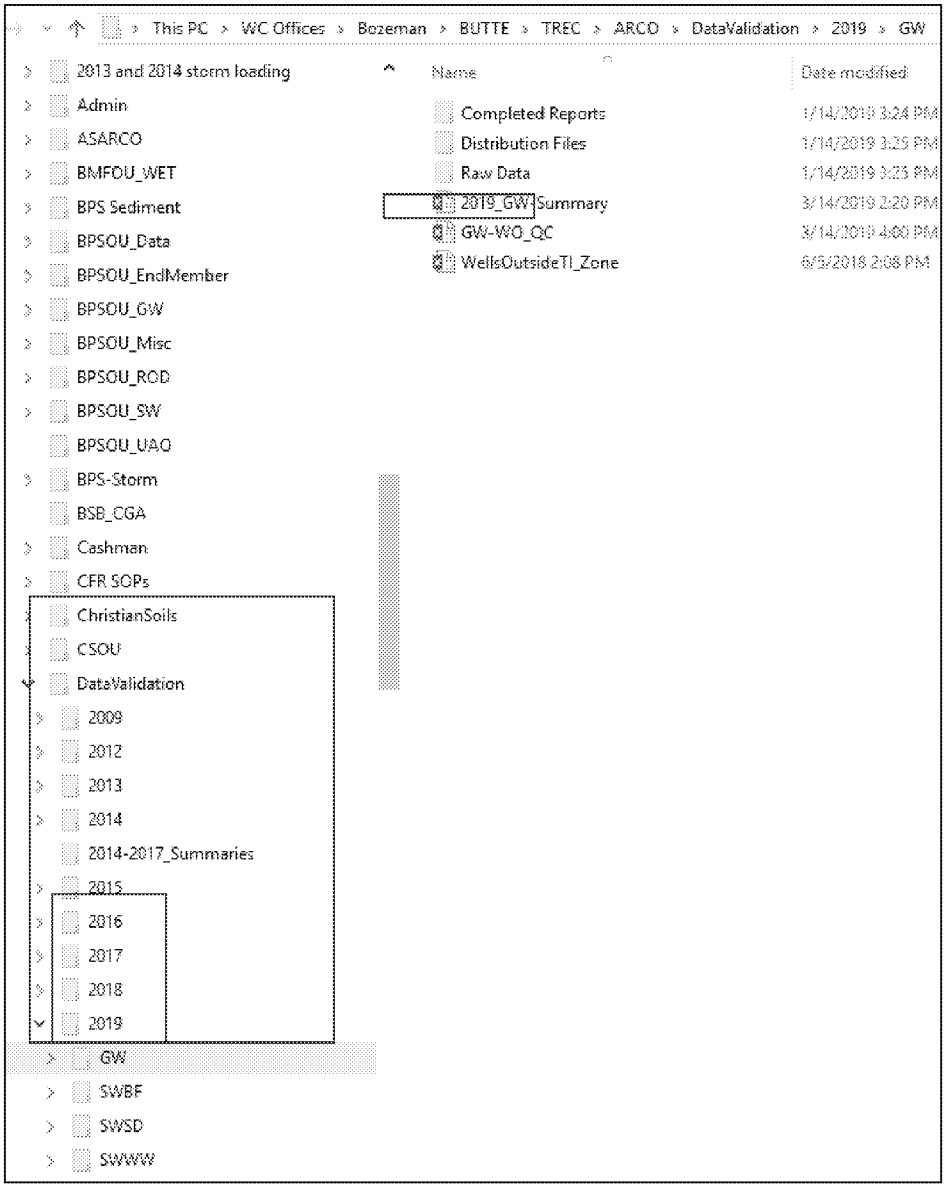
Figure 9 – Location of Individual BPSOU Field QC Files



4.1.1 Group Samples in Field QC Batches

As previously discussed, the field QC frequency for most projects is one field duplicate and one field blank per 20 primary samples. This is an overall rate; thus, if a project consists of 21-40 primary samples, two of each required QC samples could be collected on a single day. Ideally, field QC samples will be collected at a rate of one duplicate, one blank (as appropriate) within the first 20 primary samples collected. The second duplicate and blank will be collected between primary sample 21 and 40, the third QC sample set will be collected between primary sample 41 and 60, and so on. Results of the first duplicate and field blank will be used to assess primary samples 1-20, results of the second duplicate and field blank will be used to assess primary samples 21-40, etc. If all field QC samples were collected on a single day, the results of all field QC samples would be applied to all of the samples. By spacing QC samples out, the QC sample results can be applied to fewer samples. This is advantageous when field

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blank or duplicate sample results prompt qualifications. The primary sample counts (groups of 20) includes only primary samples; field QC samples are not included in the count.

Try to group primary samples/QC samples by SDG to simplify the validation process. In order to batch field QC samples with laboratory SDGs, it is allowable to group primary samples in groups of up to 22 primary samples (assuming the QC sample rate is 5% of primary samples). If this is done, some groups will have fewer than 20 samples. No matter how samples are batched, the overall field QC rate must have been met.

If multiple field QC sets were required for a single SDG, in the appropriate column of the Data validation spreadsheet (Field QC Batch), enter the field sample ID of the field blank (or other field QC sample) that aligns with each data point. This will serve as a filter to ease the data review process. In the Data Summary Table, name each tab in a manner that identifies the QC sample data that is within the tab. For example, if a field duplicate set is SW0041/SW0042, the tab containing that data could be named Dup041-042. An exact naming convention is not required, but an intuitive convention should be used.

4.2 Verify Field QC Parameters

Required field QC sample frequency and sample type is indicated in project QAPPs. Verify that QC sample type and frequency were met. All field QC samples must be analyzed for the same parameters analyzed in primary samples. If field QC sample type, frequency, or analyses did not meet the criteria specified in the project QAPP, this will be indicated on the field checklist, which is discussed in Section 5.1

4.3 Field Blank Results

Field collected blanks may be identified as field blanks, "trip" blanks, rinsate blanks, equipment contamination blanks, or cross contamination blanks. In this discussion, all of these will be referred to as FBs, and ideally all of these should have results less than the MDL. Any BP LaMP approved laboratory should report results to the MDL; thus, results below the detection limit will be reported as "< MDL" or as a value at the MDL accompanied by a "U" qualifier. If a result is reported at the MDL with no "U" qualifier, then the result was a detection at the MDL. Unless indicated in the project QAPP, laboratories not in the LaMP program may not report to the MDL, but to the reporting limit (RL). If this occurs, field blank results should be < RL.

Field collected blank results may be influenced by laboratory blank results; but, use discretion in qualifying field collected blank results based on laboratory blank detections. While the majority of projects do assess field collected blank results based on laboratory blank results, there are projects which assess the two types of blanks separately. Check with the project manager or quality assurance officer to determine which practice is followed for the analytical results being validated.

If FB results will be assessed against laboratory blanks, determine if it is likely that the FB result was influenced by laboratory contamination. With Level 3 and Level 4 packages, ICB and CCB detections should be applied to FB results only if the FB was analyzed between out of control ICB/CCB samples. Since MBs apply to entire laboratory QC batches, FB results should be assessed against MB results, assuming the FB was analyzed in the same laboratory QC batch which had MB detections. There will be instances when FB results apply to samples which were analyzed in two or more laboratory QC batches. Table 43 provides guidance on assessing sample results when this occurs. Table 44 provides guidance for

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qualifying data based on FB detections. Table 43 and Table 44 should be used together. First determine which situation in Table 43 applies, then apply qualifiers as indicated in Table 44. If a data validation qualifier is assigned for the FB detection, add reason code "FB". Refer to Section 3.3.3 Laboratory Blank Data for guidance in qualifying data for laboratory blank detections.

Unlike laboratory blanks, the laboratory instrument value is not used in assessing FB detections. Use the reported result of the FBs and associated samples in assessing FB detections.

Table 43 – Field Blank Assessment in Relation to Laboratory Blanks

FB/MB/Samples all in same lab QC batch			
FB Result	Lab Blank > MDL	FB Qualification	Associated Sample Qualification
FB < MDL	MB > FB	No qualification for FB	Qualify samples based on lab blank
FB > MDL	MB > FB	Qualify FB for MB detection	Qualify samples based on lab blank detection
FB > MDL	FB > MB	FB result ≤ 2X lab blank result, qualify FB for MB detection	Qualify samples based on FB detection
FB > MDL	FB > MB	FB result > 2X lab blank result, do not qualify FB result for MB detection	Qualify samples based on FB detection
FB/MB in same lab QC batch, FB associated samples in two or more lab QC batches			
FB Result	Lab Blank > MDL	FB Qualification	Sample Qualification
FB < MDL	MB > FB	No qualification for FB	Qualify samples based on lab blank
FB > MDL	MB > FB	Qualify FB for lab blank detection	Determine which samples were in impacted lab QC batch, if the MB was > FB, qualify the samples in that lab batch for lab blank detection. Samples which were in a lab QC batch with in-control lab blanks, qualify for FB detection.
FB > MDL	FB > MB	FB result ≤ 2X lab blank result, qualify FB for MB detection	
FB > MDL	FB > MB	FB result > 2X lab blank result, do not qualify FB result for MB detection	

Table 44 – Field Blank Action

Field Blank Result	Sample Results	Action for Samples
FB < Lab Blank	Any	No action for FB detection. Data assessed based on lab blank results
> MDL, but ≤ 1.5X MDL	Non-detect ≥ MDL	No action No action
> 1.5X MDL	Non-detect ≥ MDL, but ≤ RL > RL but ≤ 10X blank value > 10X blank value	No action Qualify results as estimated non-detect (UJ) Qualify results as estimated high (J+) No action

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Field Blank Result	Sample Results	Action for Samples
< 2X |-MDL|	Non-detect ≤ 5x absolute blank value	Qualify results as estimated (UJ) Qualify results as estimated low (J-)

4.4 Field Duplicate Results

Check the RPDs between primary and duplicate samples and assign data qualifiers as indicated in Table 45. The RPD is determined by:

$$RPD\% = \frac{|S - D|}{\frac{S + D}{2}} \times 100$$

Where S = primary sample

D = duplicate sample

Acceptable RPDs are ≤ 20% for aqueous samples and ≤ 35% for solid samples. The 20%/35% limits are applicable when both the primary and duplicate sample are ≥ 5X the RL. If either the primary or duplicate sample are < 5X the RL, an acceptable RPD is < RL for aqueous samples or < 2X the RL for solid samples. Note that qualifications based on FD RPDs are not applied to field collected blanks. Field collected blanks are made up of DI water, thus, the sample matrix differs from that which makes up the primary and duplicate sample.

When assessing RPDs, round to the whole number, with values < 20.5/35.5 rounded down and values ≥ 20.5/35.5 rounded up.

Table 45 – Field Duplicate Action

Duplicate Sample Results	Action for Samples
Both primary and duplicate sample ≥ 5X RL & RPD > 20%/35%	Qualify results ≥ MDL as estimated (J) Qualify non-detects as estimated non-detect (UJ)
Primary or duplicate sample result < 5X RL & absolute difference between sample and duplicate > RL (2X RL for solids)	Qualify results ≥ MDL as estimated (J) Qualify non-detects as estimated non-detect (UJ)
Primary or duplicate sample result < 5X RL & absolute difference between sample and duplicate ≤ RL (2X RL for solids)	No action

4.5 Check Laboratory Reported Sample Concentrations

For metals and mercury analyses in which both total and dissolved analyses have been performed, compare the laboratory reported total concentrations to the dissolved concentrations. For projects which report both total and dissolved concentrations, there is a worksheet which performs this comparison within the DVS spreadsheet. The column Rslts Diff uses the calculation (Total Result – Dissolved

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Result); thus, the difference should be positive. Check for negative differences. In the case where numerous dissolved concentrations exceed total concentrations for the same sample, a switch (either by the laboratory or sampling team) is likely. Notify the laboratory of the results and ask for a sample confirmation or rerun if possible.

5.0 QUALITY DESIGNATION

Data quality is assessed by assigning each data point a quality of Enforcement (E), Screening (S), or Rejected (R). Before assigning quality, the Field Checklist must be completed, and samples must be designated as meeting Level A or Level B criteria. Note that only primary samples are assigned a quality status. A quality status is not applicable to field QC samples.

5.1 Level A/B Assessment

Note that Level A/B applies to entire samples, not individual data points.

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Figure 10 presents an example Field Checklist. The checklist may differ slightly across projects. The

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Figure 10 example was developed for Clark Fork River Superfund Site Investigations (CFRSSI) projects; and for those projects, the checklist is often referred to as the Level A/B checklist. The checklist is fairly self-explanatory. The checklist information can be found in field logbooks or on electronic field forms. If this information is not found in the logbooks or forms, within reason, it can be discerned through conversation with the sampling team. However, if conversations are necessary, the sampling team should be instructed to document the missing information for all future field efforts.

Based on the checklist review, all samples (primary samples and field QC samples) are designated as Level A, Level B, or Unusable. If a sample receives Level A or Unusable designation, all results for that sample would be qualified as estimated (J), and the reason code AB would be assigned. It is possible for a sample to be designated as Level B, but individual data points for that sample to be qualified as estimated and coded AB. This would only happen if the field QC samples associated with that sample did not undergo the full analysis the sample underwent. For example, if manganese analysis was requested for two primary samples, but the field duplicate did not undergo manganese analysis, the field QC requirements (item III.3) would not be complete for manganese analysis. In such a case, the sample would be considered Level B, but the manganese results for the two samples would be qualified J, and an AB reason code applied.

A Level A/B checklists is attached as an appendix to this SOP, and they can also be found at the link below.

..\AB_Checklists\Level_AB_Checklist.docx

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Figure 10 – Example Field Checklist

Level A/B Screening Checklist			
I.	General Information		II. Screening Results
	Site:	SITE NAME	Data are:
	Project:	SITE NAME/LAB SDG #	1) Unusable _____
	Client:		2) Level A YES or NO
	Sample Matrix:		3) Level B YES or NO
II.	Level A Screening		
			Yes/No
1.	Sampling date		
2.	Sample team or leader		
3.	Physical description of sample location		
4.	Sample depth (soils)		
5.	Sample collection technique		
6.	Field preparation technique		
7.	Sample preservation technique		
8.	Sample shipping records		
III.	Level B Screening		
1.	Field instrumentation methods and standardization complete		
2.	Sample container preparation		
3.	Collection of field replicates (insert QAPP requirement)		
4.	Proper and decontaminated sampling equipment		
5.	Field custody documentation		
6.	Shipping custody documentation		
7.	Traceable sample designation number		
8.	Field notebook(s), custody records in secure repository		
9.	Completed field forms		

5.2 Quality Designation

Each primary sample data point is assigned a quality, Enforcement, Screening, or Rejected. Field QC samples do not receive a data quality designation. Enforcement quality data meet all QA/QC and documentation requirements and can be categorized as definitive data with unrestricted use. Screening

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quality data do not meet the applicable QA/QC requirements and/or documentation requirements and can be categorized as data whose associated numerical values are estimated. Unusable data may result from inappropriate sampling, analysis, or documentation procedures; or from field or laboratory calibration and/or QC sample results which are far outside of acceptable criteria. Unusable data is given a qualifier and a quality of R, rejected and these data cannot be used. Table 46 provides a matrix for determining data quality assignment.

Table 46 – Data Quality Matrix

Data Validation Qualifier	Level A/B Designation		
	Level B	Level A	Rejected
No qualifier or U	Enforcement (E)	Screening (S)	Unusable (R)
J, J+, J- or UJ	Screening (S)	Screening (S)	Unusable (R)
R	Unusable (R)	Unusable (R)	Unusable (R)

While it is always advisable to have eyes on every data point being validated, the formula below can be used to assign data quality. This formula assumes that data validation qualifiers are entered in column G. Column references may need to be adjusted, depending on the set-up of the DVS being used.

=(IF(OR(G2="",G2="U"),"E",IF(OR(G2="J",G2="UJ",G2="J-",G2="J+"),"S",IF((G2="R"), "R", "E"))))

6.0 DATA VALIDATION SUMMARY

6.1 Data Validation Summary

The data validation checklist that is compiled throughout the validation process is one portion of the data validation summary. A second component is a short write-up which summarizes the outcome of data validation. The summary should state the number of primary sample data points, the number (and percentage) of data points which were assessed as enforcement quality, the number (and percentage) of data points assessed as screening quality, and the number (and percentage) that were rejected. The summary should also state the reason data points did not meet enforcement quality. Refer to the checklists in Appendix D for examples of data validation summaries.

6.2 Data Assessment Report (DAR.)

The frequency that data assessment reports (DARs) must be compiled differs among projects. For BPSOU projects, the DAR is compiled annually, and submitted as an appendix to annual Data Summary Reports. Rocker OU DARs are compiled quarterly and submitted as an appendix to quarterly Operations and Monitoring Reports. The details, table formats, and checklists included in DARs differs among projects. Generally, the depth of detail is driven by the project manager in conference with the project quality assurance officer, as well as the Agency reviewer. DARs include a write-up of data validation results of all analytical data for the reporting period, several tables, and checklists. Example DARs can be found at the links below.

BPSOU Groundwater DAR

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..\..\..\..\..\..\..\Projects\TREC\9208_AR_MT_BPSOU\9208 - 2009 BPSOU\9208-003_SW-GW_Monitoring\02_Groundwater\03_DSRs\2016\Appendices\Appendix A - Data Validation\2016_BPSOU_GW_AppendixA_DQA.pdf

Rocker Groundwater DAR

..\..\Rocker\2017Rpts\Qtr4\AppG_DatValidation4QTR17.pdf

6.2.1 Review the DVS.

Once the data validation spreadsheet, the data validation checklist, and the data validation summary are compiled, a competent person should review the spreadsheet, the checklist, and the summary report. Use the following items as a guide.

1. Ensure that all requested analyses Have been reported. This can be done by performing a total count of data points. For example, a BPSOU storm drain package containing 16 samples (each analyzed for TSS, and total and dissolved (As, Cd, Cu, Fe, Hg, Pb, and Zn) should contain 240 data points (ie. 15 (analytes) X 16 (samples) = 240). If the count does not equal the expected count, first review the chain of custody. For BPSOU wet weather samples, it is not unusual that analyses will be missing due to inadequate sample volume. The COC will indicate the requested analysis. If there is nothing unexpected on the COC, consult the sample receipt form which follows the COC in the laboratory data package. This form will indicate if samples spilled en route to the lab, or at the lab. If the sample receipt form cannot explain the missing analyses, consult sample preparation records, and finally contact the laboratory project manager.
2. Make sure all required fields are populated in the distribution file: data quality, field QC Batch, modified MDL for all samples affected by MB detection, and AB designation (separate tab).
3. Perform filter checks within the distribution file to check for the following mistakes:
   a. No Non-Detects were qualified J and No detects were qualified UJ (unless the data point was qualified as "UJ" during laboratory or FB assessment.)
   b. No FB samples were qualified due to MS, MSD, LDS, SD, FD, or FB codes. Exceptions would be if the FB sample was used as the parent sample for the MS or LDS. (Note that if the FB was used as the parent for the MS, by default, it is the parent for the MSD; and, if the FB was used as the parent for the SD, then the parent sample result should be < 50X the MDL, thus the SD was not assessed for percent difference.)
4. Double check the TR vs Dis metals concentration comparison. This is performed within the distribution file under the tab "DisTR_DBLinked". If there are numerous dissolved concentrations > total concentrations for a sample, it is likely that the two sample aliquots were mislabeled in the field, or a mix-up occurred at the lab.
5. Review the checklist and compare qualifications within the checklist to qualifications in the distribution file to make sure that the three are in agreement, and to ensure that qualifiers were not entered incorrectly in the distribution file.
6. Unfilter results in the tab "forDVEntry". Scroll through sample results and perform a comparison looking for outliers or potentially mis-qualified results. Make sure all results are unfiltered prior to submittal.

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6.3 Submit the Distribution File to the Data Team.

For BPSOU projects, data tracking should be performed within the DV Index Excel sheet. Completion, reviews, any rejected results or special cases (ie. switched sample results) should be put in the Notes section of the DV Index file. This will be useful when compiling the Data Assessment Report for each Data Summary Report. Once review and any necessary revisions are made, send the distribution file with the pre-assigned naming convention to: Donna Hawley, Jonathan Longden, and the appropriate project email address:

mailto:dhawley@woodardcurran.com
mailto:jlongden@woodardcurran.com

BPSOU and Rocker data: bpsoudata@woodardcurran.com
Great Falls Data: TrecDataGF@woodardcurran.com

The naming convention differs among projects, but the return file name will be very similar to the distribution file name. Details for the return file naming convention are provided within each distribution file.

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APPENDICES

Appendix A Measurement Performance Criteria for Data

Appendix B Comprehensive Holding Time Table

Appendix C Level A/B Checklist

Appendix D Data Validation Checklists

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Appendix A Measurement Performance Criteria for Data

Measurement performance criteria are established by defining acceptance criteria and quantitative or qualitative goals (e.g., control limits) for accuracy, precision, representativeness, comparability and completeness of measurement data. The definitions of precision, accuracy, representativeness, comparability and completeness are provided below along with the acceptance criteria for data collected. Equations for calculation of precision, accuracy and completeness are provided in Table 1– Holding Times and Preservation Requirements.

Table A1 Precision, Accuracy and Completeness Calculations Equations

Characteristic	Formula	Symbols
Precision (as relative percent difference, RPD)	$RPD = \frac{(x_i - x_j)}{\left(\frac{x_i + x_j}{2}\right)} \times 100$	x_i, x_j: replicate values of x
Precision (as relative standard deviation, RSD, otherwise known as coefficient of variation)	$RSD = \frac{\sigma}{\bar{x}} \times 100$	σ: sample standard deviation $\bar{x}$: sample mean
Accuracy (as percent recovery, R, for samples without a background level of the analyte, such as reference materials, laboratory control samples and performance evaluation samples)	$R = \frac{x}{t} \times 100$	x: sample value t: true or assumed value
Accuracy (as percent recovery, R, for samples with a background level of the analyte, such as matrix spikes)	$R = \frac{SSR - SR}{SA} \times 100$	SSR: spiked sample result SR: sample result SA: spike added
Accuracy (as percent difference, D, for samples > 50X the MDL, which have undergone at least a five-fold dilution, with the result, S, corrected for the dilution)	$D = \frac{\lvert I - S \rvert}{I} \times 100$	I: initial sample result S: serial dilution result
Completeness (as a percentage, C)	$C = \frac{n}{N} \times 100$	n: number of valid data points produced N: total number of samples taken

Precision

Precision is the level of agreement among repeated measurements of the same characteristic. There are two general forms of uncertainty. The first is the random error component of the data collection process. The second is inherent stochastic variability, which cannot be eliminated but can be described.

Data precision is assessed by determining the agreement between replicate measurements of the same sample and/or measurements of duplicate samples. The overall random error component of precision is a function of the sampling. The analytical precision is determined by the analysis of field duplicates by laboratories and by replicate analyses of the same sample. An analytical duplicate is the preferred measure of analytical method precision. When analytes are present in samples at concentrations below or near the quantitation limit, precision may be evaluated using duplicate analyses of laboratory prepared samples such as duplicate laboratory matrix spike samples (MS/MSD), duplicate laboratory control spike samples

(LCS/LCSD), and/or laboratory duplicate (LD) samples. Precision can be measured as relative percent difference (RPD) or as relative standard deviation (RSD) (also known as a coefficient of variation). Formulae for both are presented in Table A1.

Accuracy/Bias

Accuracy is the degree of difference between the measured or calculated value and the true value. It is a measure of the bias or systematic error of the entire data collection process. Potential sources of systematic errors include:

- sample collection methods;
- physical or chemical instability of the samples;
- interference effects during sample analysis;
- calibration of the measurement system; and
- contamination.

Field blanks and laboratory method blanks (MB) may be analyzed to assess artifacts introduced during sampling, transport and/or analysis that may affect the accuracy of the data. In addition, initial calibration verifications (ICVs), continuing calibration verifications (CCVs), initial calibration blanks (ICBs), and continuing calibration blanks (CCBs) are used to verify that sample concentrations are accurately measured by the analytical instrument throughout the analytical run. Note that ICV, CCV, ICB, and CCB results are reported only in Level III and IV data packages.

Representativeness

Data representativeness is defined as the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point or environmental conditions. Representativeness is a qualitative parameter that is most concerned with the proper design of the sampling program. Representativeness of samples shall be achieved through the careful selection of sampling locations and methods. Sample representativeness may also be evaluated using the RPDs for field duplicate results, as well as field blank results. Agreement between duplicate samples is applicable to representativeness of individual sampling points, not the overall sampling program. If agreement between field duplicates is acceptable ($\leq$ 20% RPD for sample concentrations greater than five times the reporting limit, and a delta < the RL for samples less than five times the reporting limit), it can be assured that the reported concentration is a valid representative measure of near-aquifer conditions. If agreement between duplicate samples is not acceptable, the reported concentration must be considered an estimation of conditions.

Comparability

Data comparability is defined as the measure of the confidence with which one data set can be compared to another. Comparability is a qualitative parameter but must be considered in the design of the sampling plan and selection of analytical methods, quality control protocols, and data reporting requirements. Comparability is ensured by analyzing samples obtained in accordance with appropriate SOPs. All analytical data should be calculated and reported in units consistent with standard reporting procedures so that the results of the analyses can be compared with those of other laboratories, if necessary.

Completeness

Completeness refers to the amount of usable data produced during a sampling and analysis program. When determining completeness, also consider the number of samples that were collected in terms of the number of samples that were anticipated to be collected.

Sensitivity

Sensitivity refers to the capability to quantify an analyte at a given concentration, and this parameter is associated with the instrument and method detection limits, and the project reporting limits. The desired analytical sensitivity are typically method detection limits less than the applicable water quality standards specified in Montana Circular DEQ-7, Montana Numeric Water Quality Standards and detection limits that will allow geochemical analysis.

Appendix B Comprehensive Holding Time Table

Table B1 – Expanded List of Holding Times and Preservation Requirements

Analyte	Method	Holding Time	Preservative	BPSOU BF	BPSOU WW	Diagnostic	Expanded	BPSOU GW	Rocker	Great Falls
Alkalinity: Total, Carbonate, Bicarbonate, & Hydroxide	SM 2320B	14 days	Raw 0-6°C	Total only	Total only			x	x	
Anions by Chromatography (bromide, chloride, fluoride, sulfate)	EPA 300.0	28 days	Raw 0-6°C				Cl, F, SO4	Cl, SO4		x
Anions by Chromatography (orthophosphate-P, nitrate, nitrite)	EPA 300.0	48 hours	Raw 0-6°C							
Chloride	SM4500-Cl C	28 days	Raw 0-6°C						X	
Fluoride	SM 4500-F– C	28 days	Raw 0-6°C							
Orthophosphate-P	SM4500-P B/E	48 hours	Raw 0-6°C							
Sulfate	ASTMD 516	28 days	Raw 0-6°C	x	x	x			x	
Dissolved Organic Carbon/Total Organic Carbon (DOC/TOC)	SM 5310 C	28 days	H_2SO_4 < pH 2 0-6°C	DOC	DOC		DOC			

Analyte	Method	Holding Time	Preservative	BPSOU BF	BPSOU WW	Diagnostic	Expanded	BPSOU GW	Rocker	Great Falls
Hardness[1]	SM 2340B	180 days	HNO_3 < pH 2	x	x					
Mercury (aqueous) total and dissolved by CVAA	EPA 245.1, SW846 7470	28 days	HNO_3 < pH 2	245.1	245.1			245.1		
Metals (aqueous) total and dissolved by ICP-AES	EPA 200.7, SW846 6010	180 days	HNO_3 < pH 2				SW846 6010B			
Metals (aqueous) total and dissolved by ICP-MS	EPA 200.8, SW846 6020, 6020A, 6020B,	180 days	HNO_3 < pH 2	200.8	200.8	200.8	SW846 6020A	200.8	200.8	
Metals (aqueous) - Dissolved Exotic by ICP-MS (Cs & Rb)	SW6020A_E	180 days	HNO_3 < pH 2				x			
Nitrogen - Ammonia	**EPA 350.1** SM 4500-NH3 B/C	28 days	H_2SO_4 < pH 2 0-6°C	x	x					
Nitrogen - NO_2/NO_3	**SM 4500-NO3 H** SM 4500-NO3 E SM 4500-NO2 B	28 days	H_2SO_4 < pH 2 0-6°C	x	x					

Analyte	Method	Holding Time	Preservative	BPSOU BF	BPSOU WW	Diagnostic	Expanded	BPSOU GW	Rocker	Great Falls
Nitrogen - Total Kjeldahl Nitrogen	**EPA 351.2** SM 4500-Norg B	28 days	H_2SO_4 < pH 2 0-6°C	x	x					
pH	EPA 150.1	24 hours	Raw 0-6°C				x	x		
Solids - Total Dissolved Solids	SM 2540C	7 days	Raw 0-6°C	x	x			x		
Solids - Total Suspended Solids	SM 2540D	7 days	Raw 0-6°C	x	x	x				
Solids, Total (TS)	SM 2540B	7 days	Raw 0-6°C							
Solids, Volatile (VS)	SM 2540 E / EPA 160.4	7 days	Raw 0-6°C							
Solids, Settleable (SS)	SM 2540 F	48 hours	Raw 0-6°C							
Solids, Volatile Suspended (VSS)	SM 2540 D / EPA 160.4	7 days	Raw 0-6°C							
Specific Conductivity	SM 2510B	28 days	Raw 0-6°C				x	x		

Analyte	Method	Holding Time	Preservative	BPSOU BF	BPSOU WW	Diagnostic	Expanded	BPSOU GW	Rocker	Great Falls
Total Metals in Solids by ICP-MS (Sb, As, Ba, Cd, Cr, Cu, Pb, Mn, Mo, Ni, U, & Zn)	SW6020	180 days	None				x			
Phosphorus - **Total**/Dissolved	**SM 4500P-B**/E	28 days	H_2SO_4 < pH 2 0-6°C	x	x					
Biochemical Oxygen Demand (BOD)	SM 5210 B	48 hours	Raw 0-6°C							
Chemical Oxygen Demand (COD)	SM 5220 D	28 days	H_2SO_4 < pH 2 0-6°C							
Sulfide, Soluble	SM 4500-S2− D	15 minutes	Raw 0-6°C							
Sulfide, Total	SM 4500-S2− D	7 days	ZnAc2 & NaOH pH > 9 0-6°C							

Appendix C Level A/B Checklist

Level A/B Screening Checklist

I.	General Information		II. Screening Results
	Site/BIF:	BPSOU	Data are:
	Project:	Base Flow SW Monitoring	1) Unusable _____
	Client:	Atlantic Richfield	2) Level A YES
	Sample Matrix:	Water	3) Level B YES

II.	Level A Screening	
		Yes/No
1.	Sampling date	Yes
2.	Sample team/or leader	Yes
3.	Physical description of sample location	Yes
4.	Sample depth (soils)	N/A
5.	Sample collection technique	Yes
6.	Field preparation technique	Yes
7.	Sample preservation technique	Yes
8.	Sample shipping records	Yes

III.	Level B Screening	
		Yes/No
1.	Field instrumentation methods and standardization complete	Yes
2.	Sample container preparation	Yes
3.	Collection of field replicates (1/20 minimum)	Yes
4.	Proper and decontaminated sampling equipment	Yes
5.	Field custody documentation	Yes
6.	Shipping custody documentation	Yes
7.	Traceable sample designation number	Yes
8.	Field notebook(s), custody records in secure repository	Yes
9.	Completed field forms (COC Record)	Yes

Appendix D Data Validation Checklists